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OPTIMIZATION AND MEASUREMENT OF WATER REMOVAL ON A LABORATORY SCALE-PSA SYSTEM

SUMMARY REPORT: 13X, 3-LAYER, 4-LAYER, AND 5-LAYER PSA BEDS

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The design and operation of a laboratory-scale (100 SLPM) pressure swing adsorption (PSA) system is discussed. This work emphasizes the proper experimental procedures required for accurate and reproducible PSA system operation as well as the concept of layered adsorbent beds for optimum water removal. Results of the optimization are presented based on conditions defined by scaling down flows from a full-scale (400 SCFM) system.

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PREFACE

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1. INTRODUCTION

One current advanced air purification technology being considered for military applications is pressure swing adsorption (PSA).[1] PSA is a well accepted commercial process for air purification, usually in the area of air drying.[2] In this process, typical specifications call for the reduction of water vapor concentrations from 30,000 to 3 ppm.[3] In the current study, laboratory-scale PSA experiments are performed over a number of bed configurations in order to study the performance of water removal under a variety of conditions. This study reviews initial efforts to optimize PSA systems for water removal using operating conditions defined by scaling down flows from a full-scale (400 SCFM) system. Temperatures and concentrations are obtained from estimates of full-scale performance.

This work emphasizes two areas: (1) proper experimental procedures required for accurate measurements and reproducible PSA system operation, and (2) layered adsorbent beds for optimum water removal.

2. EXPERIMENTATION

2.1 Pressure Swing Adsorption Apparatus.

A schematic of the pressure swing adsorption (PSA) apparatus is shown in Figure 1. A cutaway view of the PSA beds is shown in Figure 2 and details the configuration of the bed assembly. High pressure air is provided from an oil-less compressor and is regulated using a mass flow controller. Water is added to the preheated air stream as steam and mixed in a large surge tank at elevated temperatures. The upstream surge tank dampens any short-term variations in the feed pressure, flow rate, and water concentration prior to entering the PSA beds. The feed stream is conditioned at the system temperature and then challenged to the PSA beds by alternately cycling the beds using four 3-way, air-actuated ball valves. This valve switching allows one bed to be challenged with feed at high pressure while the other bed is purged with a fraction of the product at low pressure. The product flow rate is set with a mass flow controller and the system pressure is regulated using a control valve on the purge line after the product-purge flow split. A more in depth procedure is described below.

There are four subsystems that comprise the main PSA apparatus, namely: (1) the feed humidification system, (2) the pressure and temperature control system, (3) the flow

control and valving system, and the (4) the humidity analysis system. All four subsystems are automated with a computer-controlled interface using National Instrument's LabVIEW programming language. The PSA data acquisition program was developed under contract at the U.S. Army Edgewood Research, Development, and Engineering Center (ERDEC) and allows the system to be fully automated for unattended operation and both monitor and measure all aspects of the system. Some of the main features of this experimental apparatus are that: (1) experiments may be performed over a wide range of humidified conditions; (2) the system is fully automated and requires only an initial operator input at the beginning of an experiment; and (3) scale-up effects from a laboratory-scale to a full-scale system may be easily examined. This apparatus also is capable of measuring the effects of humidity on the performance of a PSA system removing a toxic chemical challenge vapor.

2.1.1   Feed Humidification System.

A layout of the humidification system is shown in Figure 1. The pressurized feed air is supplied by an oil-less air compressor located in an adjacent room at a pressure of 85 - 100 psig. The compressor air is further conditioned using both refrigerated and desiccant dryers providing air with a dew point below -50 °C at atmospheric conditions. The feed air stream is humidified by injecting liquid water to achieve a desired feed dew point to challenge the PSA beds.

The water addition to humidify the feed air is a three step process. The first step requires water to be delivered under pressure and dispensed into the system using an HPLC pump (Micromeritics, Model 760) at a typical flow rate around 2 ml/min. The second step requires the water to be dropped onto a heater assembly to generate steam. The steam generation assembly consists of an insulated fire-rod (Watlow, 400W) to produce steam and is connected to a temperature controller (Omega, CN9000A) which has a temperature set point of 175 °C. The last step provides preheated air (~ 175 °C) using an insulated Watlow cartridge heater to carry the steam upward through a coalescing filter and into a surge tank to provide further mixing of the feed stream. The tank is wrapped with a heating blanket and insulated to maintain a temperature of around 100 °C using a temperature controller (Fenwal, Model 550). The surge tank is of sufficient size (38 liters) to maintain a constant pressure source and dampen any challenge concentration or flow fluctuations. The remainder of the feed air lines leading to the PSA system are insulated to prevent condensation of the pressurized and humidified air stream.

2.1.2   Pressure and Temperature Control System.

The PSA system pressure is controlled with a globe valve (Badger Meter, Inc.; Research Control Inc., Type 807) having a Cv of 0.8 on the purge side after the product-purge split. The input control pressure is monitored by a 0 - 80 psig pressure transducer (Validyne Inc., Model No. DP15-48) on the feed surge tank. Placing the control on the surge tank minimized any pressure transients that may occur in the system. The pressure transducer signal was connected to a standard pressure controller (Omega, Model CN2011) and the output current

signal (0 - 20 mA) from the controller is connected to a linear positioner. The current signal is then converted to an output control pressure (0 - 20 psig) to automatically open or close the globe valve as needed. System pressure control of 30 to 80 psig is easily attained with this configuration.

The main PSA system along with all of the beds, tubing, and valves is contained in a heated and insulated box, see Figure 1. Heat is provided by a fan and heater assembly from a Hewlett Packard 5880 Gas Chromatograph oven. The fan and heater are mounted near the base of the insulated box and are controlled by a standard temperature controller (Omega, Model 4001JC). Temperature control in the range of 30 to 80 °C is easily achieved with this design. The humidified feed stream is regulated to the PSA system temperature by using a heat exchanger made from a 50' roll of 3/8" O.D. copper tubing. This helps ensure the feed air is at or near the system temperature prior to challenging the PSA beds. Monitoring the temperature in the feed end cap shows that the challenge air temperature was ± 2 °C of the desired system bed temperature.

2.1.3   Flow Control and Valving.

The feed air flow is regulated by using a 0 - 100 SLPM mass flow controller (Tylan General Inc., FC-262/FC-2920V) upstream of the feed surge tank and steam generation system. The product air flow is also controlled with a 0 - 100 SLPM mass flow controller. Both the feed and product mass flow controllers are connected to a flow control box (Tylan General Inc., Model RO-28) allowing the user to dial in flow set points. During the transient pressurization and depressurization steps, the air pressures, temperatures, and flow rates are quickly changing. These transients appear to affect the product air flow more than the feed air flow since the product controller is downstream of the system.

Since the flow controllers respond and control on much longer time scales than the length of the PSA blowdown and pressurization steps (<0.5 sec), it is fair to assume that the mass flow controllers are acting like fixed-position valves. Therefore, although it is difficult to measure the flow rates absolutely because of the pressure fluctuations, an average flow may be more representative of the flow rate through the controller. The average flow rates are measured using a calibrated, dry gas totalizing meter. A total integrated volume of air over a specified period of time gives flow rates that are within ± 5% of the readings from the mass flow controller. The flow rates used for the experiment are those measured with the totalizer, and are recorded both prior to starting an experiment and at its conclusion.

The humidified challenge air is switched between both sets of PSA beds using four, 3-way air-actuated 3/8" trunion valves (Whitey, SS-83XTS6-51DC). Pneumatic actuation provided rapid response of the valves to change the flow path of the feed and purge streams. The valves are packed with teflon seals to provide long cycle life and minimize any leakage. The valve configuration and sequencing is provided by SPDT relays located on the data acquisition module controlled through the computer interface.

2.1.4   Humidity Analysis System.

The feed and product humidity are monitored continuously throughout the course of the experiment. The feed is analyzed by taking a slip stream of air through an orifice which at a system pressure of 65 psig delivers approximately 2 LPM. A vacuum pump is set to draw 0.5 LPM from this air stream through a dew point hygrometer (EG&G Inc., Model 911) to analyze for the feed water content. Feed dew points measured in this system are typically around 25 °C at atmospheric pressure which requires heat tracing the analysis lines to prevent possible condensation.

The product dew points are much lower than the feed with values ranging from 0 to -70 °C and require the use of another dew point/frost point hygrometer (EG&G Inc., Model 300). This analyzer requires an external cooling bath (5 °C) for the low frost points measured in the product stream. Downstream of the product controller, a 0.5 LPM slip stream is sent to the moisture analyzer for measurement. The dew points for both the feed and product streams are sent to the data acquisition board and continuously recorded.

2.1.5   LabVIEW Data Acquisition Program.

The entire PSA program is fully automated under the LabVIEW programming language (National Instruments) and is used to both control and monitor all aspects of the system in real time. The entire control program is executed using a single multi-tasking Macintosh computer. The data acquisition system uses National Instrument's Signal Conditioning Extension for Instrumentation (SCXI) hardware. The specific components used in the system are: (1) the NB-MIO-16XL-18, a high performance multifunction board with analog (sixteen 16-bit ADCs with voltage inputs, two 12-bit DACs with voltage outputs), digital (eight lines of TTL-compatible digital I/O), and timing I/O (three 16-bit counter/timer channels) capabilities for the Macintosh NuBus; (2) the SCXI-1001, a 12-slot chassis to add plug-in boards and modules to control analog input/output signals, digital input/output signals, relays, or other DAQ board signals; (3) an SCXI-1100 module, 32-channel multiplexing amplifier with an attached SCXI-1300 general-purpose terminal block with software programmable gain settings that allows the user to record a variety of volt and millivolt signals; (4) an SCXI-1180 feedthrough panel with an attached SCXI-1302 terminal block that extends the I/O signals of the plug-in data acquisition board to the front of the SCXI chassis and allows the user to send output analog control voltages to various devices; (5) an SCXI-1120 module, 8-channel isolation amplifier with an attached SCXI-1328 isothermal, high accuracy-terminal block which allows the user to record thermocouples or other millivolt signals on eight channels and configure the gain settings for each channel independently; (6) an SCXI-1124 module, 6-channel isolated digital-to-analog converter and an attached SCXI-1325 terminal block, which allows the user to output either 0 - 10 V DC voltage signals or 0 - 20 mA current signals (software configurable) to control process devices; and (7) two SCXI-1160 modules, 16-channel SPDT digital relay with attached SCXI-1324 high-

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voltage terminal blocks that allows the user to control valve orientations, switch general purpose signals, and activate devices.

The LabVIEW data acquisition system for the PSA program allows the user to: (1) record all temperatures and pressures from the system apparatus and display data continuously to the operator; (2) record both feed and product dew points and all flow rates continuously; (3) control all timing and sequencing of the 3-way valves to direct the flow path for switching the beds; (4) change cycle times of the system while running to study the effect of cycle time on system performance; (5) control all valve sequencing for the in-bed GC sampling system when used; (6) select the GC run time, retention time, and tolerance window for chemicals as well as collecting all integration data when used; (7) select the desired in-bed ports to sample when the sampling system is used; and (8) select when to start or stop the experiment. Figure 3 shows a sample of the front panel of the PSA program used as the operator interface, and Figure 4 shows a small portion of the wiring diagram for the main program.

2.1.5.1   Temperature and Pressure Measurement.

The thermocouple probes, 1/32"-Type T, ungrounded (Omega Inc., TMQSS-032U-6) are placed in the center of the PSA bed and fixed using 1/16"-316 SS Swagelok fittings with teflon ferrules. The thermocouples could be positioned every 2.5 cm along the bed depending on where the user wished to observe the temperature swings. The millivolt signals from the thermocouples are then recorded real-time using the PSA LabVIEW program running on a Macintosh computer and the data acquisition hardware (SCXI-1120) discussed earlier. Temperatures in both the feed and product end caps are saved along with at least two in-bed temperatures and the insulated system box temperature.

Transient, in-bed pressure profiles for both the pressurization and blowdown steps are measured and recorded on the PSA system by using a pressure transducer (Validyne Inc., Model DP15-48) on the feed end of the bed. The pressure drop across the beds is also measured using a differential pressure transducer (Validyne Inc., Model DP15-28) connecting the feed and product end caps. These pressure transducers are calibrated using a calibrated reference pressure gauge. Both pressure transducer signals are sent to a Validyne pressure box (Validyne Inc., Model MC1-333) and the output voltage signal (millivolt) is sent to the SCXI-1120 module to be recorded real-time on the computer and also to the pressure controller to regulate the system pressure.

2.1.5.2   Humidity and Flow Measurement.

The feed and product dew points as well as the feed and product flow rates are typically recorded every five cycles for the results presented here. However, this value may be easily changed before or during the experiment. The voltages from both the flow control and dew point hygrometer boxes are 0 - 10 V signals and are sent together to a module (SCXI-1100) different from the thermocouples (SCXI-1120, millivolt signals) to prevent any signal

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interference with gain switching. By keeping the signals separate we are able to get a cleaner signal throughput for both devices. These signals are monitored real-time and give the operator a sense of how the system is performing.

2.1.5.3 Valve Configuration and Timing.

The PSA LabVIEW program controls all timing and sequencing of the four, 3-way valves to direct the flow paths for switching the beds every half cycle. SPDT relays in the SCXI-1160 module provide the required signal for the 24V DC solenoids on the air-actuated valves. Although not used in this experiment, a multi-port sampling system capable of collecting six samples simultaneously is connected to the system and is able to provide concentration profiles and material balances to evaluate system performance. Details of this multi-port sampling system are presented elsewhere.[4] The relay signals for both electrically activated solenoids and valves are wired into one SCXI-1160 relay module, while the positioning valves and stream selectors (based on TTL logic) are wired to a separate relay module to prevent possible signal interference.

2.2 PSA Bed Design and System Construction.

A schematic of the PSA adsorption beds is shown in Figure 2. The bed is constructed from 304 SS to a length of 40 cm with 1/8" wall thickness and has 5 cm long end caps. The inner diameter of the PSA bed is 1" and has a porous bronze disk to contain the adsorbent and allow maximum flow throughput. Sample ports for concentration and temperature probes are tapped to 1/16" NPT and drilled at 5 cm intervals along the length of the bed to permit multiple sampling. Sample ports are also drilled at 2.5 cm intervals along the adsorbent section to permit additional in-bed sampling probes.

All tubing for the system is made from 316 stainless steel to prevent corrosion from water at the high operating temperatures and pressures. The tubing size is 3/8"-O.D. to allow maximum flow through the system and minimize any pressure drop. Typical flow rates examined in this system have ranged from 70 - 100 SLPM. There are two aerosol/particulate filters (Matheson Inc., Model 6124-P12FF) placed upstream of the PSA beds. The first filter is placed after the steam generation system to collect and filter any water droplets that may be sent to the feed surge tank. The second filter is placed after the surge tank and before the bed inlet to prevent any rust particles from the tank (untreated steel) from entering the beds. On the product line, a filter (Matheson Inc., Model 6164-P4FF) is added to prevent any potential dust from fouling the product flow controller. Although the flow controllers themselves have some filtering capability, they are coarse and designed for much larger particles.

2.3 Materials.

The adsorbate used in this study is water which has been doubly distilled and deionized. The five adsorbents used in the experiments are: (1) Alumina F-200 (Coastal

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Chemical, 7x14 Mesh), (2) Coarse Silica Gel 40 (Grace-Davison, 10x12 Mesh), (3) Fine Silica Gel 40 (Grace-Davison, 18x25 Mesh), (4) 13X Molecular Sieve (UOP, HP-PSA02, 16x20 Mesh), and (5) BPL Carbon (Calgon Carbon Corp., 12x30 Mesh). Table 1 shows a tabulated listing of the adsorbents studied along with the names used in this report.

2.4 Experimental Procedure.

The experimental procedures utilized in this study will be described here. Four 3-way valves control the gas circulation path through the main PSA system. The valves are wired together so that the valve control to reverse the flow direction is simply a matter of switching a single relay. The humidified feed stream is conditioned at the system temperature and challenged to the PSA beds by cycling the valves. The product flow is regulated and the system pressure is controlled by a valve after the product-purge split. A detailed procedure is described below.

2.4.1 Adsorbent Drying.

Prior to loading a PSA bed with adsorbent, the adsorbents must be dried according to standard procedures to ensure that the adsorbent weight is consistently measured. Alumina, silica gel, and BPL carbon are heated overnight at 110 °C using a conventional drying oven to desorb any water. For 13X molecular sieve, a much more stringent heating procedure is followed because of the strong water adsorption properties. The 13X is dried overnight at 300 °C under a 3 LPM dry air flow. The drying is accomplished in a temperature-controlled tube furnace fitted with a quartz tube. The 13X is then cooled to ambient temperature and quickly added to storage bottles with leak tight lids. The other three adsorbents are stored in similar fashion. Once the adsorbents have been properly dried they are ready to pack in the PSA beds.

2.4.2 Bed Packing.

Before packing a new PSA bed, all fittings, tubing, and end caps are cleaned and inspected for damage or dusting. The feed end cap is replaced and a spacer and porous metal disk are inserted to fix the adsorbent bed position in relation to the tapped holes. The desired mass of all adsorbents is set aside for packing the beds. Next, the adsorbent is slowly added and the bed is lightly tapped to ensure even packing of the adsorbent materials. The bed depth of each individual adsorbent layer is recorded so that a measure of length and mass are kept for each material. The thermocouples (1/32"-Type T) are inserted into the bed once the desired port has been filled with adsorbent. At least two thermocouples are added for in-bed temperature sampling. After the bed is filled with the desired amount and depth of adsorbent, a final overall bed length is recorded. A second porous metal disk is positioned into the bed, and a specially sized spacer and spring are added to ensure that a one inch spring compression is applied to the bed. This was done to prevent the adsorbents from moving or fluidizing during system operation.

After the beds are packed, they are pressurized to ~ 80 psig with clean house air and then suddenly depressurized to blowdown the adsorbent. This step is repeated 30 times to try and remove any fine particles added during the loading procedure. Although the PSA system has filters on the product line, this blowdown step helps to minimize any possible dust that may be deposited in the product line and foul the flow controller.

2.4.3   Diagnostic Check.

The packed beds are then loaded into the system and all fittings are securely tightened. The thermocouples and pressure transducers are connected and a manual valve utility program is started on the computer to ensure that all wires have been properly fastened. Prior to starting an experiment the system is pressurized to 75 psig and leak checked. The four, 3-way PSA system valves are pressure checked as well to ensure that there is no bypass or leakage through the valves. Once everything has passed the initial system check, preparations are made to begin the experiment.

2.4.4   Operating Procedures.

2.4.4.1   System Start-Up.

The following sections outline the procedures followed for start-up, operation, and shut-down of the PSA system. First, the insulated cover is placed on the PSA system box and the temperature controllers are turned on to regulate the PSA box and feed surge tank temperatures. The system is placed in the bypass mode and the feed mass flow controller is set to the desired flow rate (adjusted later). The air preheater is turned on and the preheat temperature (~ 175 °C) is set by adjusting the voltage using a Variac. In bypass, the pressure is adjusted to the desired system pressure with a needle valve and monitored until stable. The feed flow rate is measured with a dry gas totalizer meter and the feed controller is adjusted until the feed flow is within ± 0.1 SLPM of the desired flow.

2.4.4.2   System Operation.

After the feed is set, the main PSA LabVIEW program is started and all initial system data is entered into the computer. In order to properly operate the PSA system, the following parameters are set: (1) full cycle times, (2) data display and record intervals, (3) data and log filenames, (4) calibration parameters for the thermocouples, and (5) start and stop times. These values tend to vary and are adjusted as necessary depending on the adsorbents and the experimental conditions. The pressure controller is set to the desired system pressure, the product flow controller is turned on, and the bypass valve is flipped to direct the flow over the PSA beds. At this point, the PSA system is operational with dry air. The product flow rate is measured with the dry gas meter and adjusted with the product flow controller until the product is within ± 0.1 SLPM of the desired flow. The purge flow is measured in a similar fashion and all three flow rates are recorded.

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The next step outlines the addition of the humidified air stream to the PSA system. The steam generator is turned on with a set point set of 175 °C. Once the steam controller has achieved this temperature, the HPLC water pump is turned on to the desired water flow to flash the water off as steam. Both the feed and product EG&G dew point hygrometers are turned on to begin recording values to the computer. At this point, the PSA system is fully operational and the performance of the system is constantly monitored with the temperatures, pressures, and flow rates being recorded by the user on data sheets and also by the computer program.

2.4.4.3   System Shut-Down.

The PSA system cycles until periodic-state is achieved at which time all flows (purge, product, and feed) are again measured and recorded. Since both the feed and purge streams have significant amounts of water that may contribute to the measured flow rate, these streams are passed through a heat exchanger in an ice bath and then through a coalescing filter to trap condensed water. This air flow is then sent to the dry gas meter to measure the flow rate. Once all three flow rates have been measured and recorded, the system is shut-down. This is accomplished by stopping the LabVIEW program and turning the HPLC pump off. The feed dew point is monitored and once it falls below 0 °C the steam generator, pressure controller, product flow controller, feed and product EG&G, box heater, and tank heater are all turned off. The system is then put into bypass and depressurized to approximately atmospheric pressure. The air preheater is turned off while the feed controller is left on to cool the system. All computer files are then saved to disk for data reduction.

3.   RESULTS AND DISCUSSION

3.1   PSA Experiments.

All PSA experiments were run until a periodic steady-state was achieved for the given set of operating conditions. Periodic steady-state was assumed when the in-bed temperature profiles repeated the same temperature path from cycle to cycle and the measured product dew point remained constant. The time to achieve periodic steady-state varied with each experiment and was highly dependent on the initial state (water loadings) of the PSA beds when the new operating conditions were set. The direction periodic steady-state was approached, i.e., from a wet state or a dry state, greatly affects the transient time. Factoring in these conditions, the periodic steady-state for an average experiment was reached in approximately 4 hours. A list of the PSA lab-scale bed configurations showing the amount of each adsorbent and its location in the bed is shown in Table 2. The lab-scale conditions that identify the flow rates and temperatures used for all the different experiments run is shown in Table 3. The flow rates for all experiments were calculated from the Case 2 conditions and were scaled based on various full-scale bed diameters for a 1" lab-scale PSA bed so that the same superficial fluid velocity was

attained for each bed. It was found during the course of the experiments that in order to get meaningful results, flow rates had to be constantly measured throughout the course of the experiment. It was found that the mass flow controllers used in the experiments tend to produce varying results from experiment to experiment and that small errors in the purge or product flow rates can result in a relatively large change in the product dew point.

3.1.1 PSA Concentration Profiles.

The PSA experiments were performed on four types of adsorbent bed configurations: (1) All 13X, (2) 3-Layer (Alumina/Coarse Silica/13X), (3) 4-Layer (Alumina/Coarse Silica/Fine Silica/13X), and (4) 5-Layer (Alumina/Coarse Silica/Fine Silica/13X/BPL). Each bed configuration lists the adsorbents in order from the feed end through the product end. Table 4 lists the names of all lab-scale experiments that were run along with the dates, conditions, and periodic steady-state product dew point results. Figure 5 through Figure 32 show the results of all the PSA experiments in terms of product dew or frost point versus cycle number, along with the feed dew point and feed and product flows recorded for the experiment. The product dew points were measured after the product flow controller with either a Shaw Dew Point Sensor or a Model 300 EG&G hygrometer. The results measured with the EG&G chilled mirror analyzer to measure frost points were found to be much more accurate than the Shaw Dew Point Sensor based on calibration results.

3.1.2 PSA Pressure Profiles.

During the course of the experiment one could change the Purge/Feed (P/F) ratio by either changing the product flow or the purge back pressure. Figure 33 shows the transient pressure profile for an experiment with a 16 sec full cycle time. Two cycles are shown with no back pressure and show the pressure at the feed end of the bed as well as the differential pressure across the bed. The data indicates that it takes about one second to pressurize the beds, but the depressurization is nearly instantaneous. Because the pressurization and depressurization steps are on such short time scales it was assumed that the system operated in a two-step process. Figure 34 shows the transient pressure profiles for an experiment with a 16 sec full cycle time and 3.5 psig purge back pressure. As can be seen by the pressure profiles, the pressurization step again takes about one second, but the depressurization step now takes about two seconds and is not nearly as efficient as the experiment with the lower purge pressure.

3.1.3 PSA Temperature Profiles.

During the course of the PSA experiments, transient temperature profiles are measured to examine the system behavior over several cycles. Temperature profiles were recorded for a 3-Layer Bed PSA experiment and are shown in Figures 35 - 38. Temperatures were measured in the feed and product end caps and also in each individual adsorbent layer. The ambient system temperature was also measured. Figure 35 shows a typical transient temperature response for two 16 sec cycles using a dry air feed. Figure 36 shows the temperature swings and

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large temperature rise resulting from the immediate addition of the humidified feed air into the system. Figure 37 shows the temperature transients after the humidified feed air has been flowing into the bed for 50 cycles (13.3 minutes). Figure 38 shows the temperature profiles after the system has attained its periodic steady-state.

3.2   13X PSA Beds.

The first set of lab-scale PSA experiments was performed on beds filled entirely with 13X. The mass of 13X was approximately 43 g and the overall bed depth for these experiments was kept constant at 12.0 cm. These experiments were scaled to simulate a 10.8" full-scale diameter bed and were the initial experiments performed to validate the PSA system. The product dewpoints were measured with the gold dot Shaw sensor that recorded dew points in the range of -30 °C to +20 °C. Figures 5 - 11 show the results of these experiments and show a variation in the results from -11.1 °C to -3.4 °C. The calibration on the gold dot sensor was applied to the actual Shaw sensor readings in order to generate the dew points. The feed, product and purge flow rates were only measured for one experiment (13X_9/4/96) during the course of this initial study. This experiment had a dew point of -8.2 °C which is about the average of all the experiments examined. During the course of another experiment (13X_9/16/96) the product flow rate was measured and found to be about 2 SLPM too low, thus the purge flow rate was 2 SLPM too high. This experiment showed a product dew point of -9.1 °C which was nearly 1 °C drier in the product dew point than the 13X_9/4/96 experiment. This result is consistent with the increased purge flow rate.

3.3   3-Layer PSA Beds.

The next set of experiments was performed using 3-Layer PSA beds. Table 2(b) shows the bed configuration and amounts of adsorbent used in this study. The beds were packed with alumina in the feed end, coarse silica gel in the middle, and 13X in the product end. This bed configuration was chosen in order to swing the high concentrations of water with the alumina and silica gel in the feed end and allow the 13X to swing the low concentrations of water on the product end. The total adsorbent mass was 46.4 g and the overall length of the bed was 12.0 cm. These experiments were scaled to simulate a 12.2" full-scale diameter bed and were the second set of experiments performed in the lab-scale system. In all of these experiments, the feed, product and purge flow rates were carefully measured at the beginning and end of each experiment, and sometimes during the experiment when time allowed. Figures 12 - 19 show the results of these experiments where a number of condition changes have been made including (1) increased or decreased product and purge flow rates, and (2) increased purge back pressure.

The initial set of experiments shown in Figures 12 -15 was performed using lab-scale PSA system #1 and was the first to show the importance of accurately measuring the purge, product and feed flow rates. These experiments show that if one is off in the measurement of the product flow by 4 SLPM that the resulting product dew point may be off by as much as 20 °C. Experiments were performed that varied the product flow rate and measured the resulting

product dew point. These experiments measured dew points using both the red dot and gold dot Shaw Meter dew point sensors and had the proper calibration applied in order to generate the appropriate dew points. The next set of experiments shown in Figures 16 -17 was performed on the second lab-scale PSA system (#2). The results from these experiments were measured with the more accurate EG&G chilled mirror sensor and show the same trend in terms of sensitivity as the other lab-scale system. A final set of PSA experiments was performed on the same 3-layer bed configuration except the majority of the dead volume was in the product end instead of the feed end. The configuration of the PSA beds for these experiments was termed "inverted" and are shown in Figures 18 -19. Also, in the Al_Si_13X_1/9/97 experiment, the influence of purge back pressure on system performance was examined. A purge back pressure of 1 psig was applied to the system and the product dew point increased by nearly 3 °C, and then a 3.5 psig back pressure was applied to the system and the product dew point increased by at least 15 °C. The 3.5 psig back pressure experiment did not come to a periodic steady-state so the results can only be evaluated in a qualitative fashion.

Using the results from the 3-Layer PSA lab-scale experiments, a sensitivity analysis may be generated that relates the product frost/dew points to the purge to feed ratio (P/F), where P/F is based on absolute column volumes of air. Before we could generate these ratios, we needed to quantify the purge back pressure using the purge flow rates through the system. Figure 39 shows the results of the pressure profiles for the product end, feed end and vent/outlet measured for the 3-Layer PSA beds as a function of purge flow rate through the system. These profiles help to characterize the pressure drops across the bed and through the 3-way outlet valve in the system. The pressure measurements were done without the cycling of the PSA system and were measured with a pressure transducer (0 - 5 psig), pressure gauge (0 - 14 psig), and water manometer (0 - 2 psig). The purge inlet pressure (product end) can now be characterized as a function of purge flow and allow an approximation of the P/F ratio as:

$$\text{Approximate Purge to Feed Air Ratio} = (F_{purge} \bullet P_{feed})/(F_{feed} \bullet P_{purge}) \tag{1}$$

Where F_{purge} and F_{feed} are the purge and feed flow rates (SLPM), respectively; and P_{purge} and P_{feed} are the purge and feed absolute pressures (psia), respectively.

This equation is only an approximation since the blowdown and pressurization gas volumes are not included. There is a significant amount of dead volume in the PSA beds that must be accounted for to obtain an accurate P/F calculation. Every half-cycle requires a certain amount of feed gas to pressurize the feed-end dead volume. That same amount of gas must be counted as blowdown gas and not pure purge flow alone. Remember, we are totalizing the purge flow <u>after</u> it leaves the bed, thus this feed-end gas is not purged. The amount of flow that is lost to the pressurization of the PSA beds is a function of the half-cycle that is run and becomes quite significant under the conditions of the experiment that we are running (8 sec half-cycles). The current 1" I.D. lab-scale beds have a dead volume of approximately 0.120 liters in the feed end that must be pressurized each half-cycle. The amount of additional feed required to pressurize this dead volume is approximately 3.8 SLPM. This is quite significant and shows the actual

conditions used in the 3-Layer PSA experiments were not the ICD-Case 2 conditions, and in fact, are actually much lower P/F ratios. With this in mind, we increased the amount of feed flow into the system as well as the purge flow in order to get a proper material balance. Thus, the exact purge-to-feed ratio for an 8 sec half cycle may be calculated as:

$$\text{Exact Purge to Feed Air Ratio} =$$
$$[(F_{purge} - 3.8) \bullet P_{feed}] / [(F_{feed} - 3.8) \bullet P_{purge}] \tag{2}$$

This ratio will tend to give lower than expected P/F ratios unless the proper amount of feed (3.8 SLPM higher) is dialed into the mass flow controller. Figures 40 - 42 show the results using the calculations that were mentioned above. Figure 40 shows the influence of accounting for the purge back pressure alone and not assuming that the system is at atmospheric pressure. This figure has the calculation from Equation 1, but does not account for the additional feed flow needed for pressurization and blowdown. The curves from Figure 40 show that by not accounting for the purge pressure results in an error of approximately 10 °C in the product frost point. Figure 41 shows the corrected P/F ratio and shows the smaller P/F values that are calculated with Equation 2. It also identifies the fact that we are running lower P/F ratios than the actual ICD-Case 2 condition. The actual P/F ratio for ICD-Case 2 should be around 1.6 which would put the standard results if we follow the curve (off-scale) to around -38 °C for the product frost point for the 3-Layer PSA beds. Figure 42 shows the results for the vapor pressure of ice as a function of the P/F ratio and shows the exponential influence of the vapor pressure with temperature.

3.4   4-Layer/5-Layer Optimized PSA Beds.

        The next set of experiments was performed using 4-Layer and 5-Layer PSA beds. Table 2 (c,d) shows the bed configuration and amounts of adsorbent used in this study. The 4-Layer PSA bed had the exact masses in the table for the adsorbent used (total mass = 48.5 g) and the bed depth was 12.5 cm. This standard 4-Layer bed differed from the 3-Layer bed in that a fine silica layer was added next to the 13X layer, which brought the ratio of coarse silica/fine silica in this bed to about 2/1. The addition of fine silica had been shown in earlier experiments to improve the water removal performance of the PSA beds.[*] Since the 5-Layer bed experiments used the 4-Layer configuration as a basis, only the initial configuration is listed in this table (Table 2d) and shows the removal of 6 g of 13X and the addition of 6 g of BPL carbon at the product end in order to keep the total bed mass at 48.5 g.

        The 4-Layer bed configuration was chosen as the standard bed and the basis for comparison in the results of this optimization study. The motivation for adding a carbon layer to the product end of the bed was to produce a PSA bed capable of producing low product dew points and providing chemical protection. The goal of the 5-Layer bed optimization study was to produce a bed that could give as low a product dew point as the 4-Layer bed. The

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[*] Unpublished data, October 1996.

configuration of the 5-Layer beds was alumina in the feed end, coarse silica gel in the middle (next to alumina), fine silica gel (next to 13X), 13X, and BPL carbon in the product end. The total adsorbent mass was fixed at 48.5 g and the overall length of the bed was approximately 12.5 cm. These experiments were scaled to simulate a 12.5" full-scale diameter bed and were the last set of experiments performed in the lab-scale system. In all of these experiments, the feed, product and purge flow rates were carefully measured at the beginning and end of the experiment. Figures 20 - 32 show the results of these experiments and show a number of condition changes with the increased or decreased product and purge flow rates as well as increased purge back pressure.

3.4.1 4-Layer PSA Beds.

The basis of the 5-Layer bed optimization experiments was generated from the 4-Layer bed experiments. In order to more efficiently run the experiments in the optimization study, two identical sets of PSA beds were run to ensure that the same results were recorded for each set of beds. Figures 20 and 21 show the results using the 4-Layer beds. The first two experiments produced results for the 4-layer beds with product dew points of about -42 °C. These two different beds produced results that were nearly identical and showed the two sets of stainless steel PSA beds to be identical. All experiments done in the optimization study were run with the beds inverted, i.e., the dead volume in the product end. These experiments also ran conditions that did not correct for the additional feed flow necessary for the pressurization step and blowdown step, thus the P/F ratio for all of the optimization experiments will be lower than the actual ICD-Case 2 conditions. Table 5 shows the results from both the 4-Layer and the 5-Layer PSA beds and shows the results in a format that one can follow the exact mass of adsorbent used in each experiment.

3.4.2 5-Layer Optimized PSA Beds.

The 5-Layer bed optimization study was performed in a systematic fashion in order to find which adsorbent was most critical in terms of water removal performance. In order to produce a bed that had chemical protection, 6 g of 13X was removed and 6 g of BPL carbon was added to the product end. This was the initial experiment performed in the study. It was shown that removing over half of the 13X and adding the mass as carbon had a detrimental effect on the water- removal performance. The product frost point increased by nearly 12 °C. Thus, a set of experiments was run that: (1) replaced a portion of alumina with 13X, (2) replaced a portion of coarse silica with 13X, and (3) replaced a portion of both alumina and silica with 13X. The mass of carbon in all of these beds was kept at 6.0 g, and the total mass of the beds was kept constant at 48.5 g. Table 5 shows a listing of all experiments performed in this optimization study along with the results and masses of adsorbent used in the experiment. Some experiments were repeated to check the validity of the results and also one experiment was performed to quantify the effect of increased purge pressure on the product dew point.

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3.4.2.1   Alumina Removal.

The first set of experiments observed the effect of removing 25% and 50% of the mass of alumina and adding the additional mass as 13X in order to keep the overall bed mass at 48.5 g. Figures 23 and 24 show the results of the removal of 25% and 50% of the alumina, respectively. Removing 25% of the alumina resulted in a decrease in the product dew point of about 2 °C to -33.5 °C. However, removing 50% of the alumina resulted in a dramatic decrease of the product dew point of over 10 °C. Thus, from this first study alone it appears that the removal of water may be greatly improved by removing the alumina in favor of 13X. There is also a potential trade-off with an increase in pressure drop due to the smaller 13X particles, but the trend towards less alumina/more 13X is quite apparent.

3.4.2.2   Silica Removal.

The next set of experiments demonstrated the effect of removing 15% and 25% of the mass of coarse silica and adding the additional mass as 13X in order to keep the overall bed mass at 48.5 g. Figures 25 and 26 show the results for removing 15% and 25% of the silica, respectively. The percentages of silica removed were chosen in order to keep the absolute amount of 13X in the bed as close as possible to the amount used in the previous alumina removal experiments. Removing 15% of the coarse silica resulted in a decrease in the product dew point of about 6 °C to -37.3 °C. However, removing 25% of the silica did not result in a large decrease in the product dew point. In fact, the product dew point decreased by only about 1 °C more to -38.4 °C. This behavior was much different than observed earlier with the removal of the alumina. It appears that there is not much difference in the trade-off by removing additional silica gel. In fact it may be detrimental with the increased pressure drop from the smaller 13X particles. It appears that one may improve performance by removing a small amount of silica gel. However, one does not greatly improve performance by removing too much silica gel.

3.4.2.3   Alumina and Silica Removal.

The next experiment removed equal amounts of both the alumina and coarse silica (3 g each) in order to bring the mass of 13X (11.6 g) to that of the 4-Layer bed keeping the overall bed mass at 48.5 g. Figures 27, 29, and 30 show the results of the removal of 3 g each of the alumina and silica. This was the first set of experiments to be duplicated and showed a product dew point of about -40 °C. During these last two experiments, the beds started off partially wet and approached the periodic state from both the "wet" and "dry" sides in both experiments. It appears that we did improve performance of this bed by removing a small portion of the alumina and silica to maintain the amount of 13X at 11.6 g, i.e., the same mass as the 4-Layer bed.

The next experiment was the first attempt at optimizing the 5-Layer beds. After examining the results from the previous experiments it was decided to remove 50% of the alumina and 15% of the silica which would boost the amount of the 13X layer to 12.9 g (up 1.3 g from

the 4-Layer Bed) while maintaining the overall bed mass at 48.5 g. Figures 28, 31, and 32 show the results of this experiment with the optimized alumina and silica removal. These experiments showed an overall product dew point of around -42 °C which is nearly identical to the 4-Layer bed result. Thus, it appears that one is able to achieve better water removal performance by adding 13X and removing an equal mass of alumina and coarse silica gel. The last experiment, shown in Figure 32, also shows the influence of a 2 psig purge back pressure. The product dew point increased nearly 17 °C with this extra back pressure that reduced the purge/feed ratio and also lowered the water removal efficiency of the PSA bed. From this study it appears that one may greatly improve system performance by removing both alumina and silica gel.

3.5   Summary.

PSA experimental results were measured over 4 different bed configurations: (1) All 13X, (2) 3-Layer bed, (3) 4-Layer bed, and (4) 5-Layer beds. In each of the experiments, a variety of different flow conditions was explored to simulate the performance of a full-scale PSA system with different bed diameters. The flow rates for the lab-scale experiments were scaled to a 1" bed diameter by keeping the superficial velocities (feed and purge) the same as the full-scale system. It was determined that measurement of the feed, product, and purge flow rates is critical in achieving the overall product dew point results. A correction to the feed flow rate and the purge flow rate must be made in order to account for the gas needed to pressurize the dead volume during each half-cycle and is critical for the short cycle times used in the lab-scale PSA experiments. A 5-Layer bed for chemical protection was configured to produce product dew points as low as the product dew points with the standard 4-Layer bed. Both the 4-Layer and 5-Layer beds have equal total masses of adsorbents.

4.   CONCLUSIONS

PSA experimental results for the removal of water under case 2 ICD conditions were measured over 4 different bed configurations: (1) All 13X, (2) 3-Layer bed (Alumina/Coarse Silica/13X), (3) 4-Layer bed (Alumina/Coarse Silica/Fine Silica/13X), and (4) 5-Layer bed (Alumina/Coarse Silica/Fine Silica/13X/BPL).

Feed, product, and purge flows must be accurately measured in order to obtain meaningful results.

Additional feed flow for pressurization and blowdown of the dead volume in the lab-scale PSA beds must be accounted for to accurately calculate the P/F ratio.

Chemical protection with the addition of BPL is possible while still maintaining good water removal performance through the use of a 5-Layer adsorbent bed.

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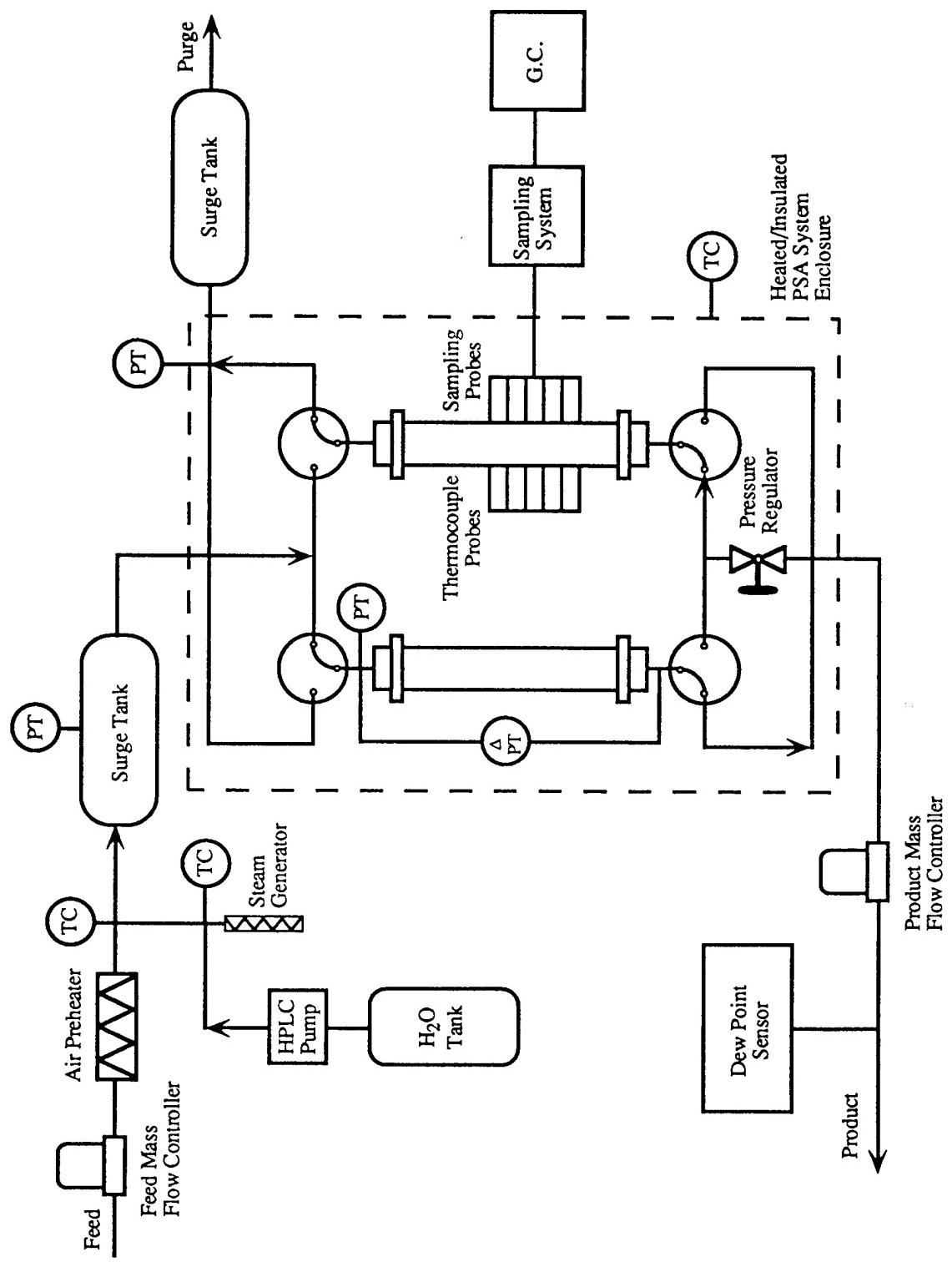
**Figure 1.** Laboratory PSA System Schematic
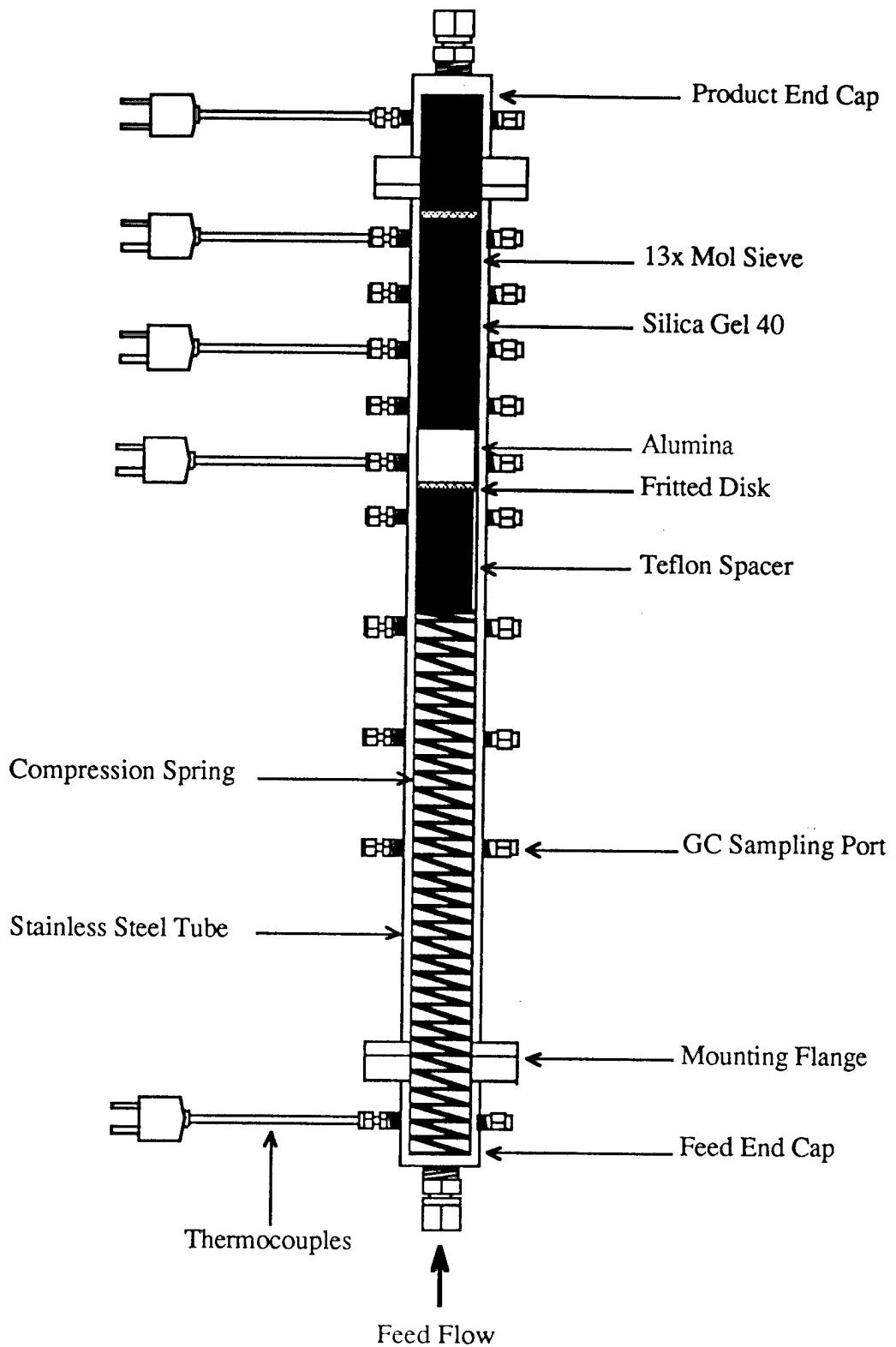
**Figure 2.** Laboratory Scale PSA 3/4-Layer Bed Configuration
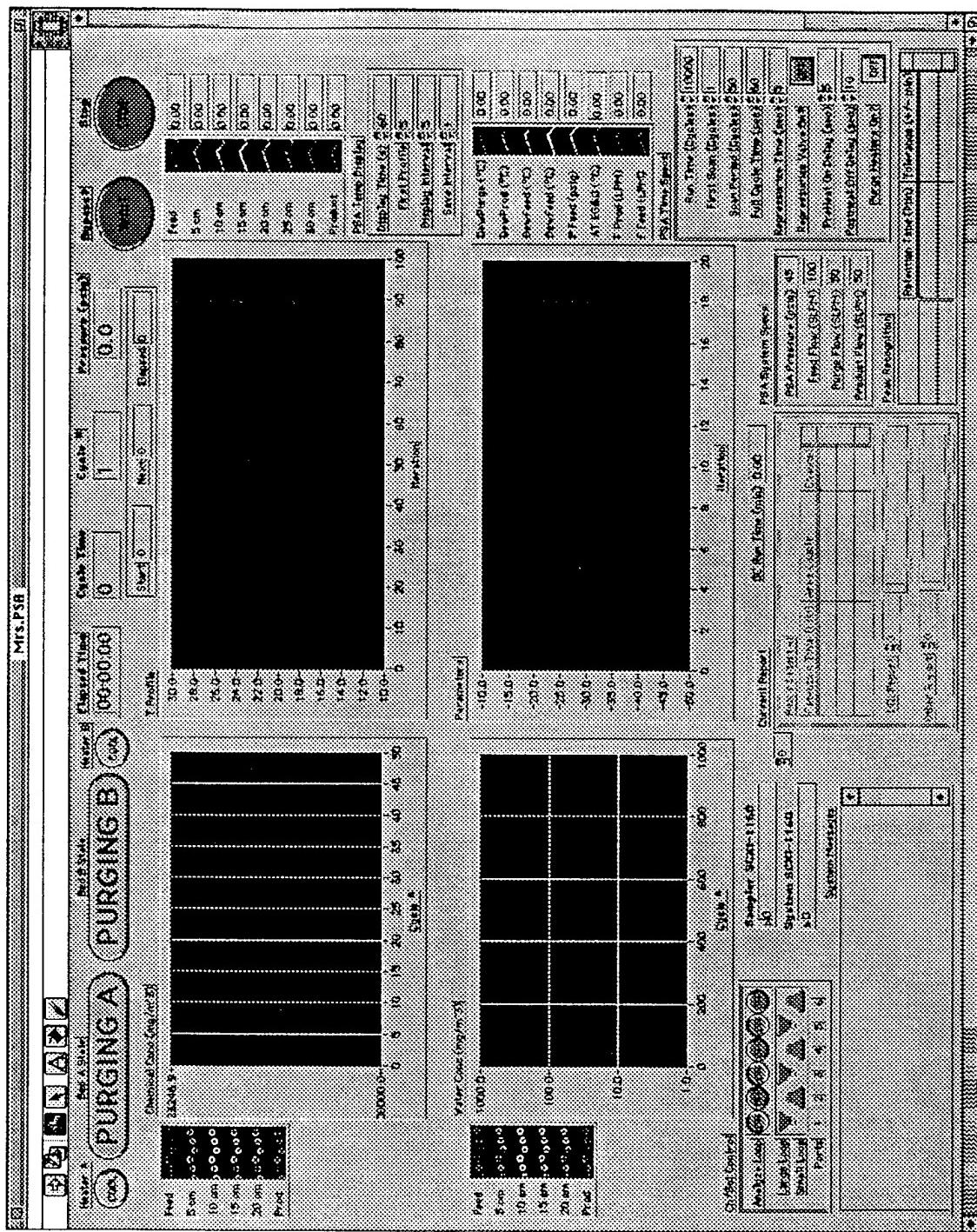
**Figure 3.** Front Panel (User Interface) PSA Program Using LabVIEW Data Acquisition Software
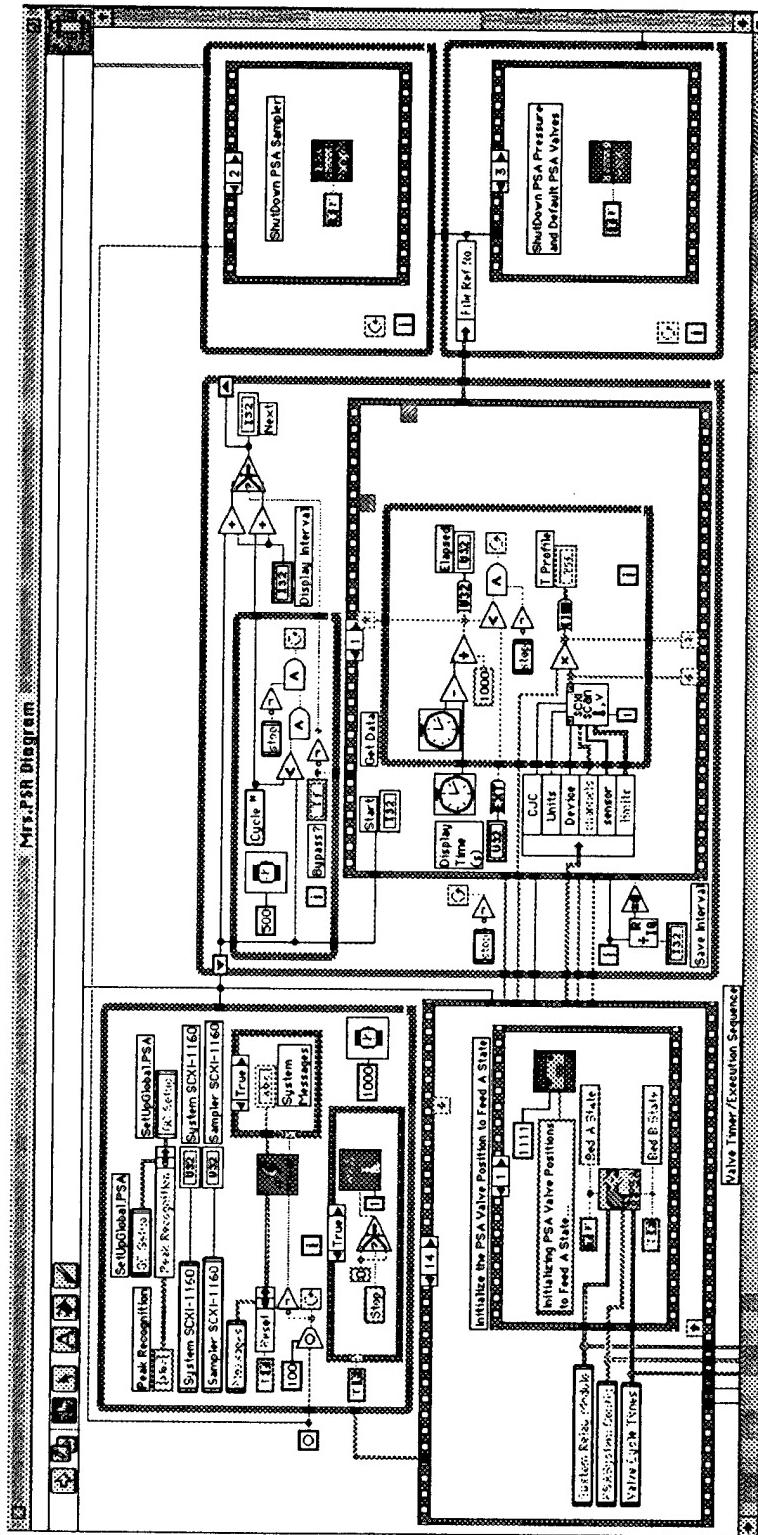
**Figure 4.** Block Diagram (Wiring Diagram) PSA Program Using LabVIEW Data Acquisition Software
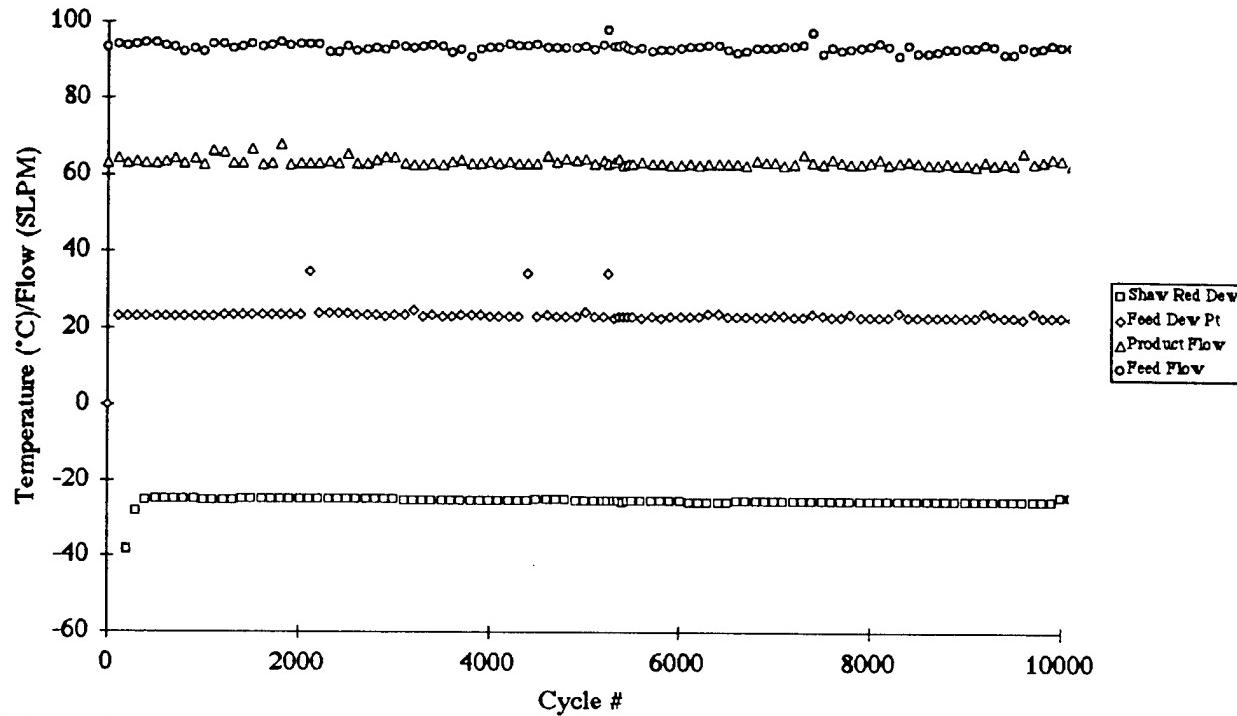
**Figure 5.** PSA Parameters (Feed and Product Flows/Dew Points), Expt-13X_8/28/96
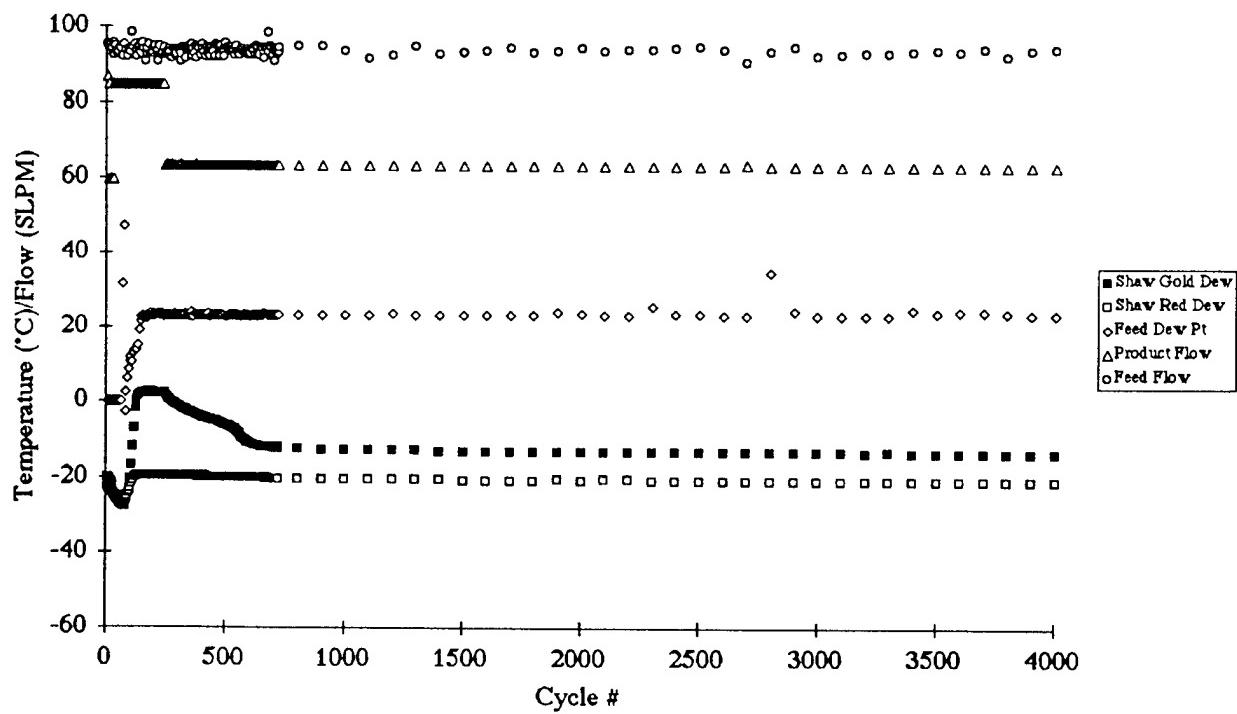
**Figure 6.** PSA Parameters (Feed and Product Flows/Dew Points), Expt-13X_9/4/96

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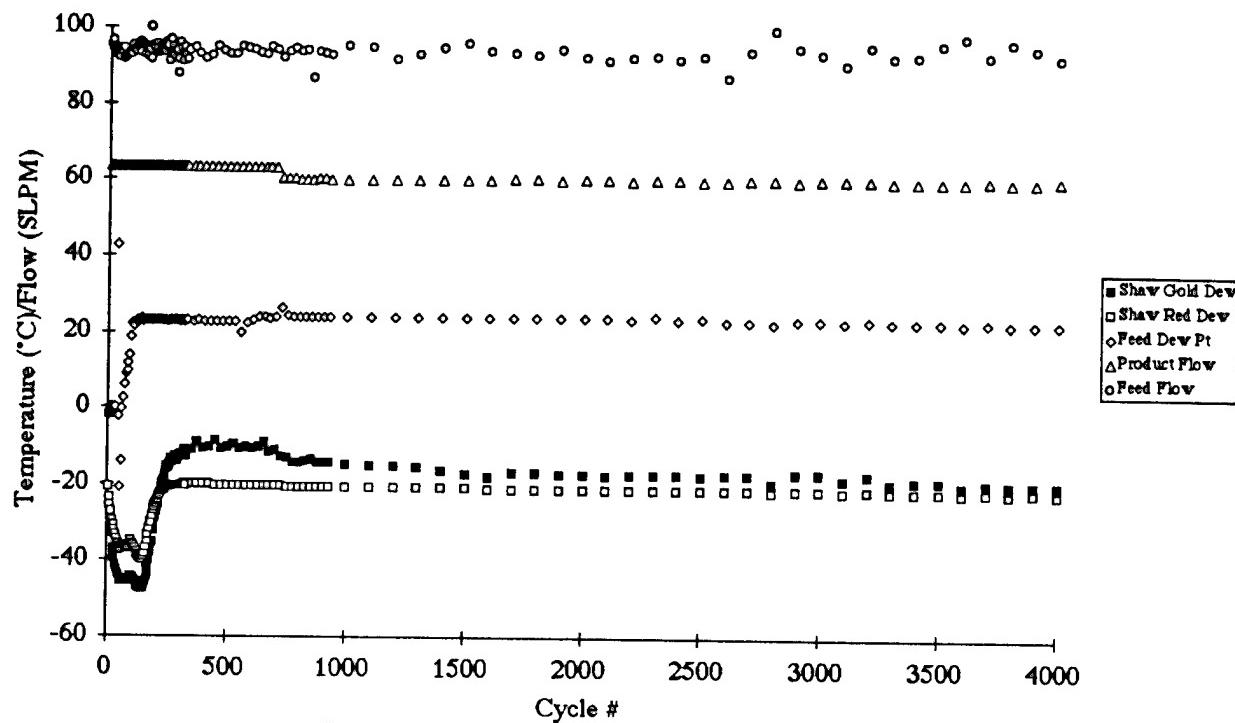
Figure 7. PSA Parameters (Feed and Product Flows/Dew Points), Expt-13X_9/10/96
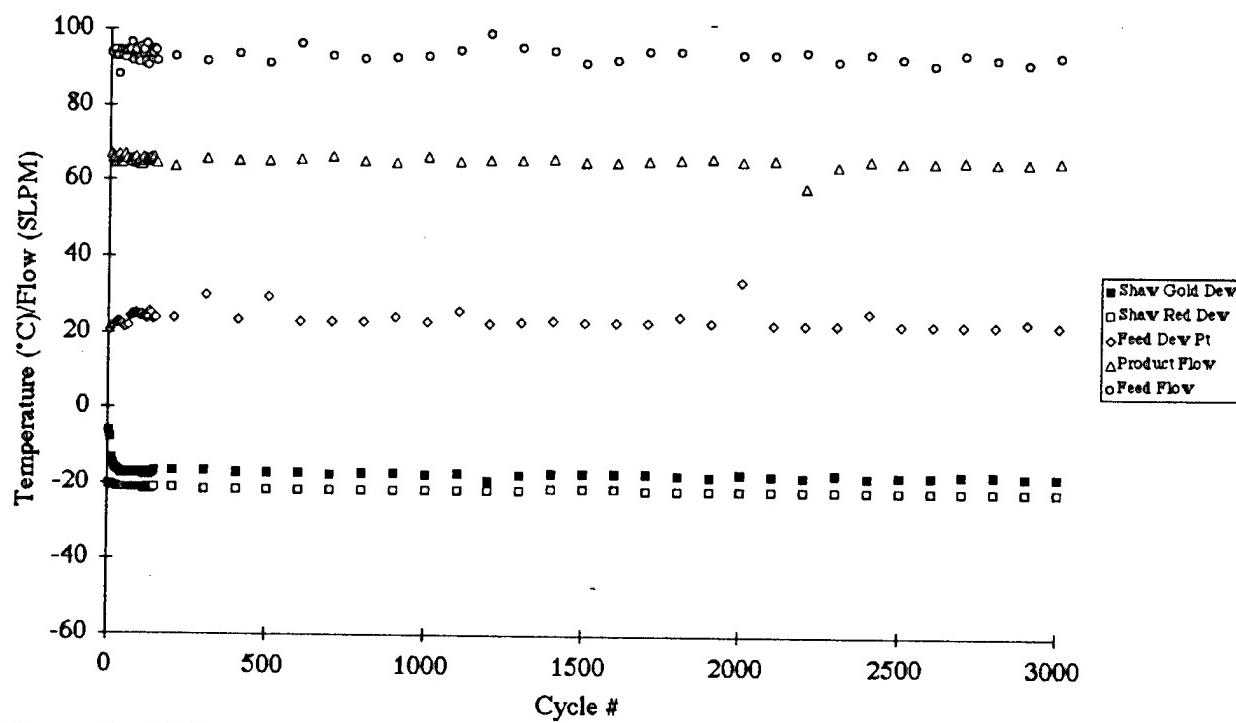
Figure 8. PSA Parameters (Feed and Product Flows/Dew Points), Expt-13X_9/11/96
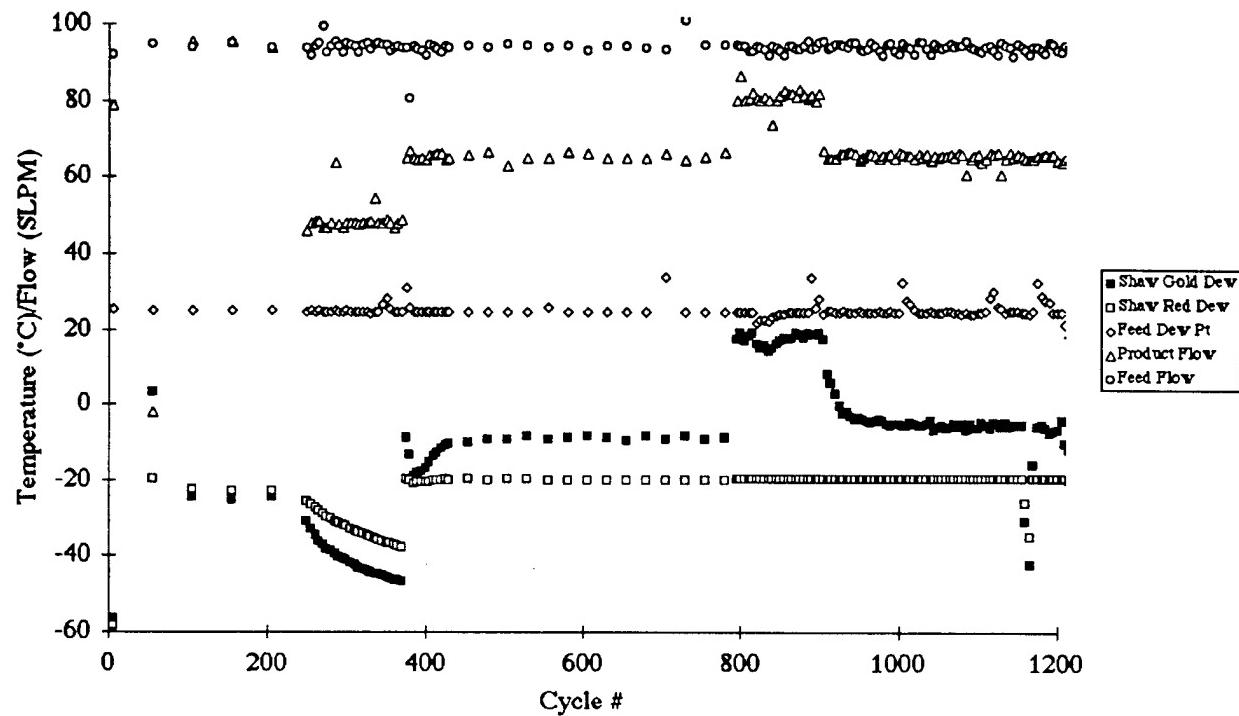
Figure 9. PSA Parameters (Feed and Product Flows/Dew Points), Expt-13X_9/13/96
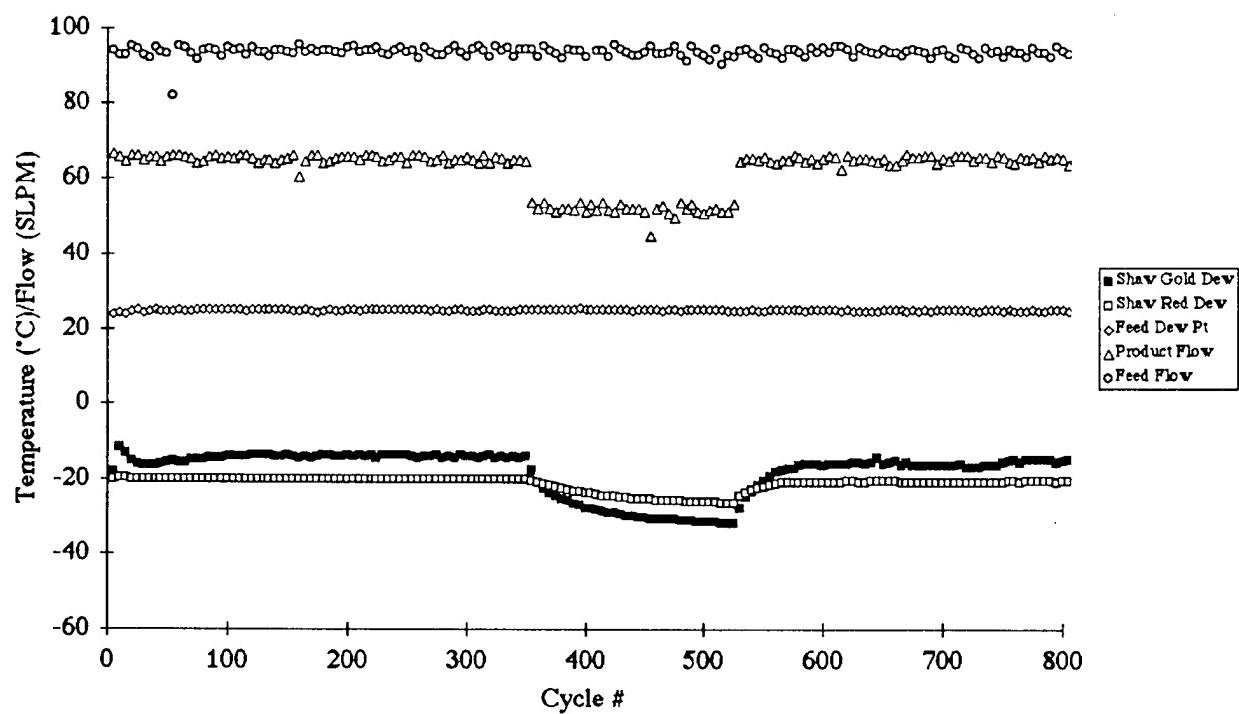
Figure 10. PSA Parameters (Feed and Product Flows/Dew Points), Expt-13X_9/16/96

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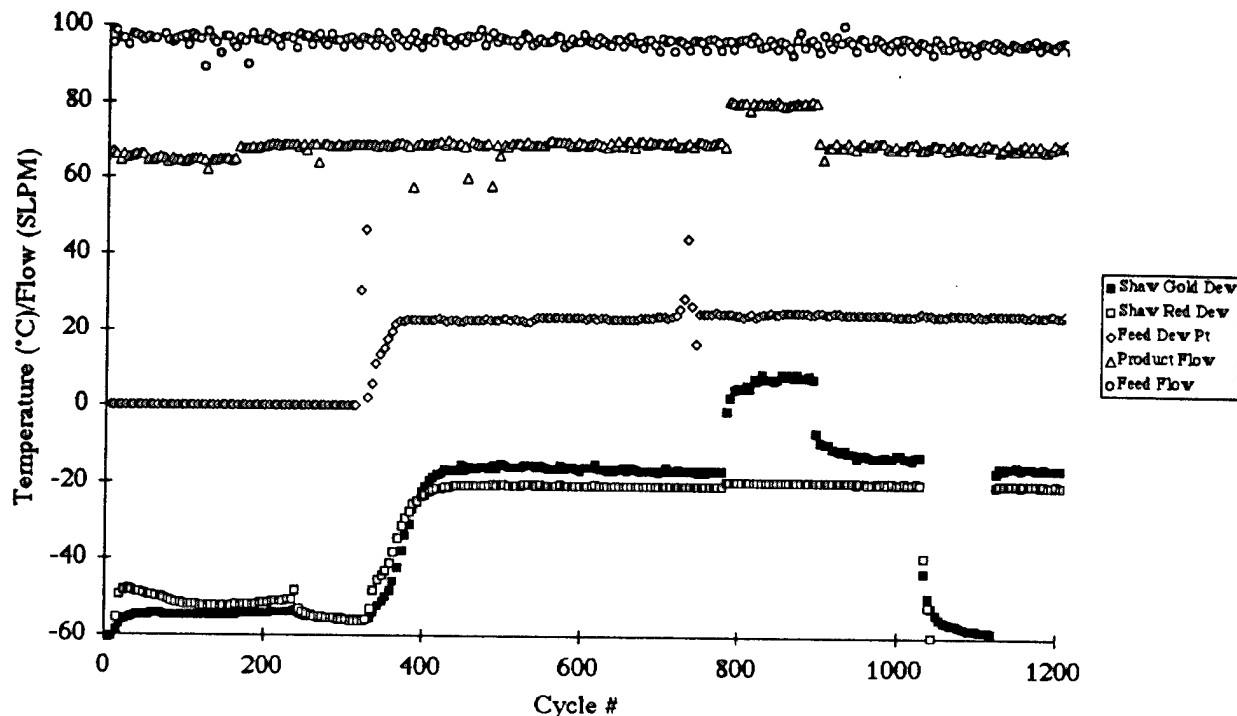
Figure 11. PSA Parameters (Feed and Product Flows/Dew Points), Expt-13X_9/19/96
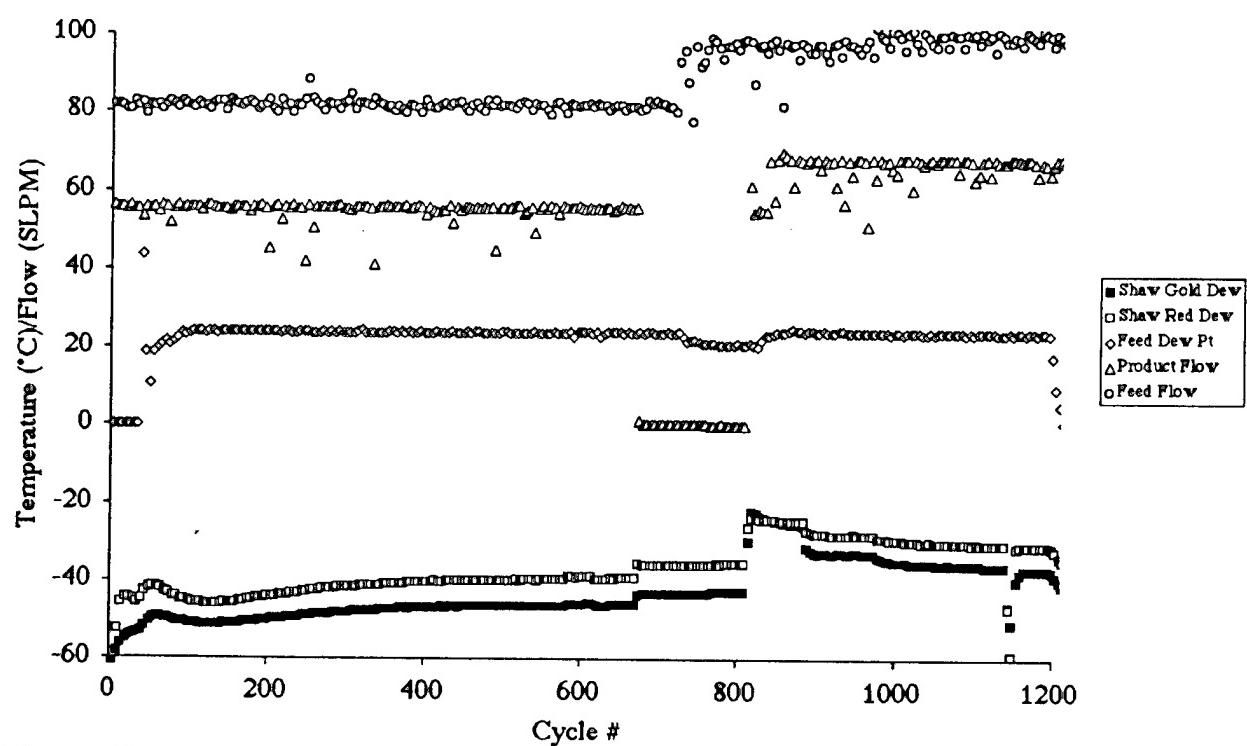
Figure 12. PSA Parameters (Feed and Product Flows/Dew Points), Expt-3-Layer_10/4/96
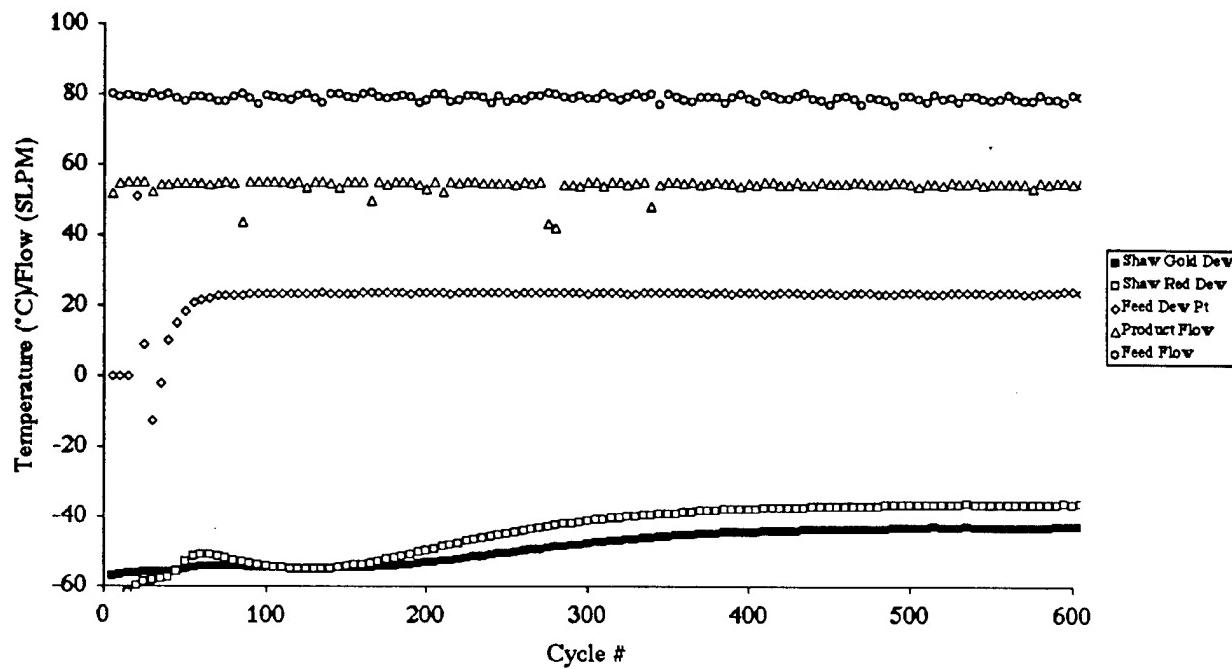
**Figure 13.** PSA Parameters (Feed and Product Flows/Dew Points), Expt-3-Layer_10/16/96
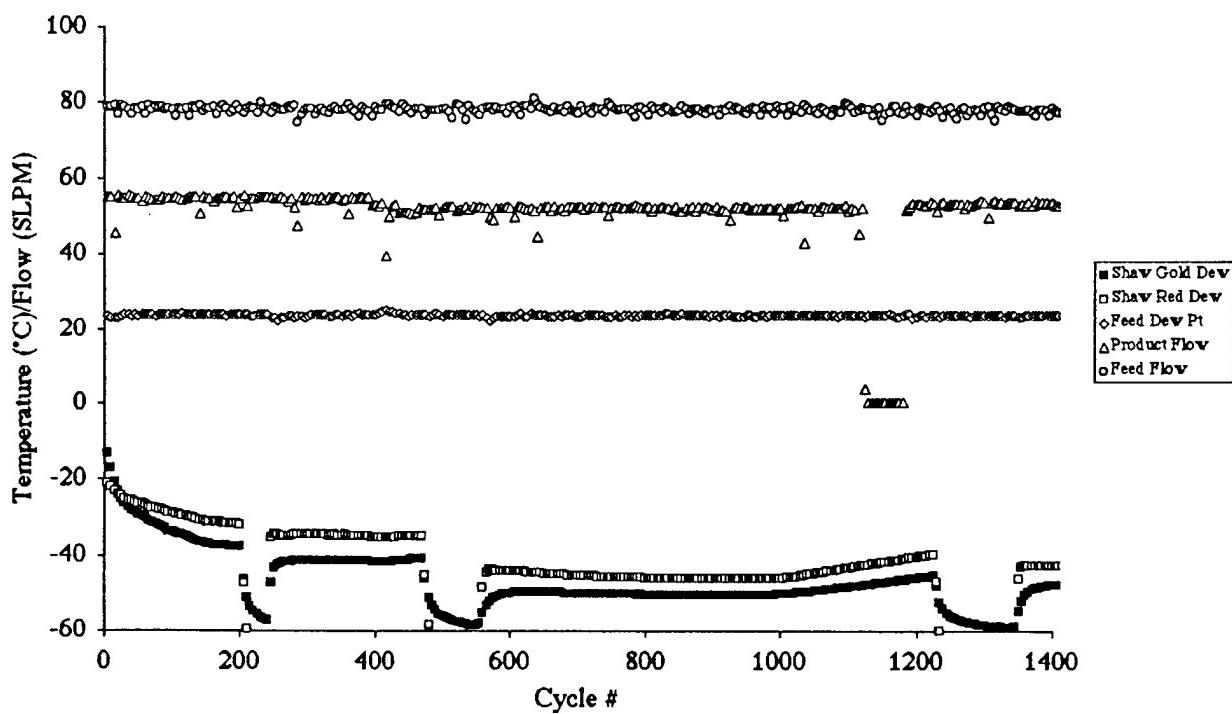

Figure 14. PSA Parameters (Feed and Product Flows/Dew Points), Expt-3-Layer_10/17/96
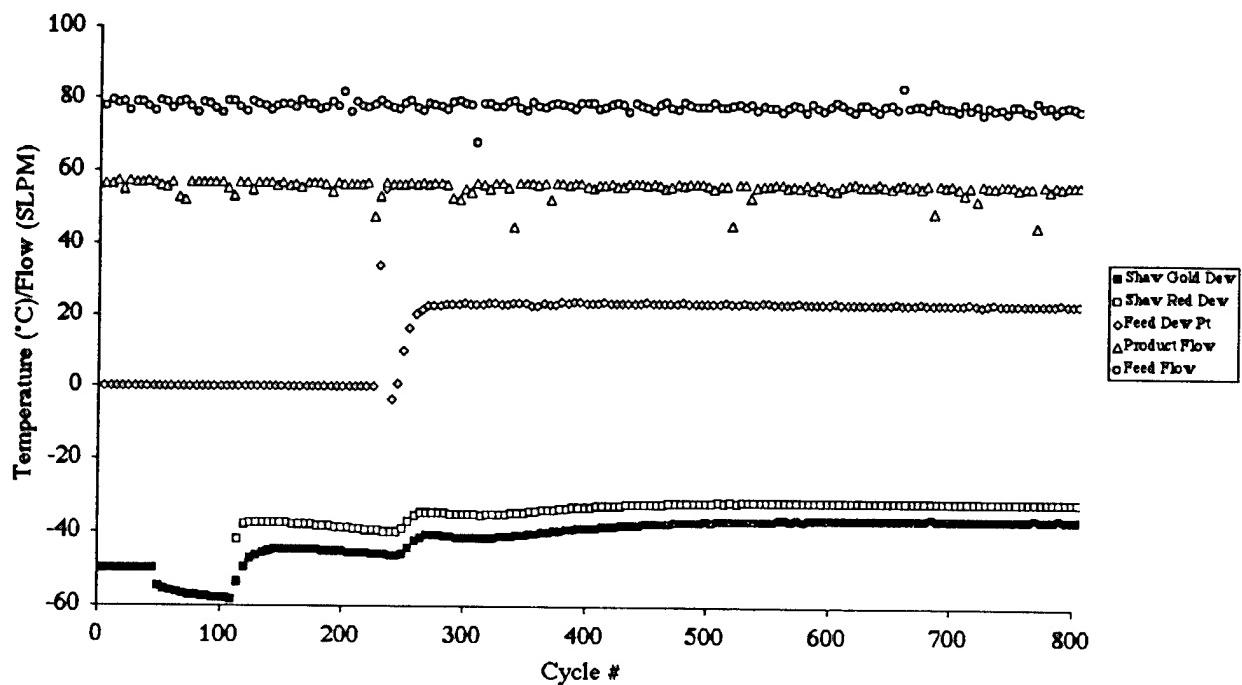
Figure 15. PSA Parameters (Feed and Product Flows/Dew Points), Expt-3-Layer_10/18/96
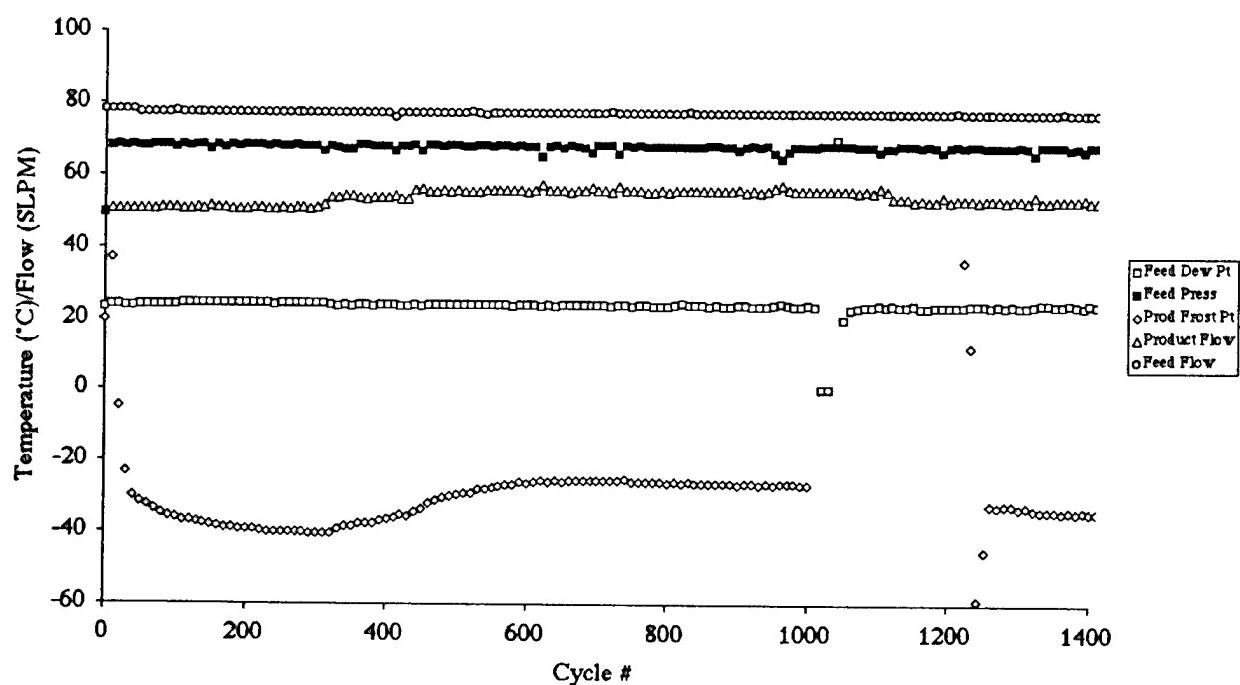
Figure 16. PSA Parameters (Feed and Product Flows/Dew Points), Expt-3-Layer_10/23/96
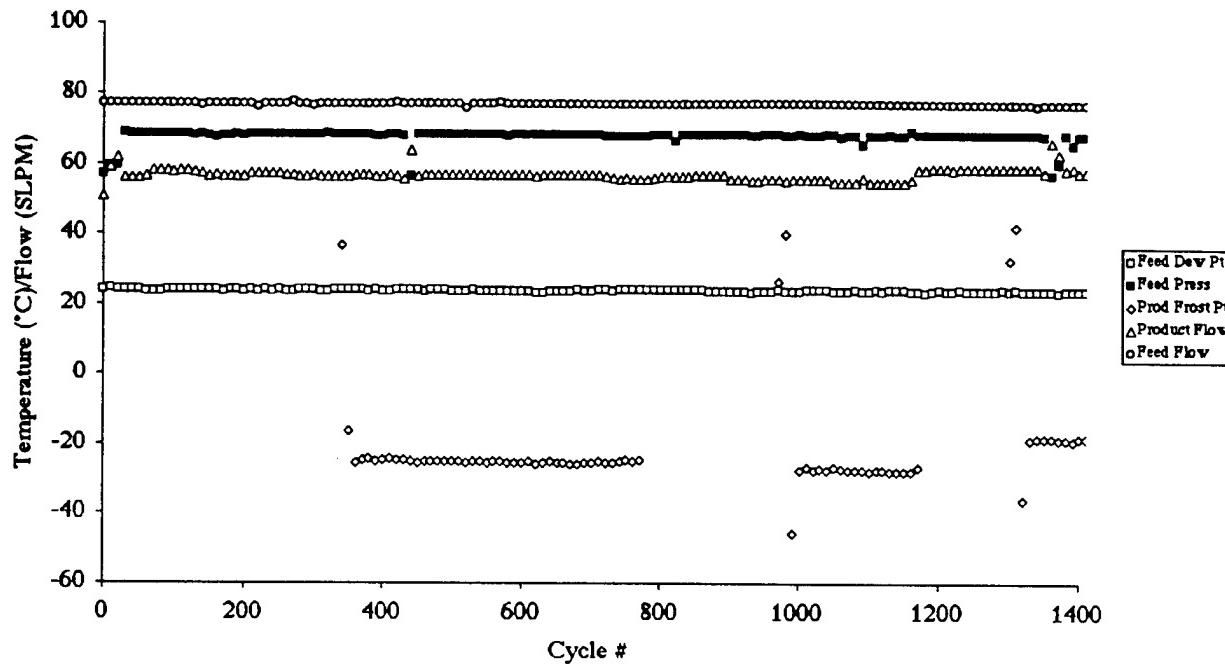
Figure 17. PSA Parameters (Feed and Product Flows/Dew Points), Expt-3-Layer_10/24/96
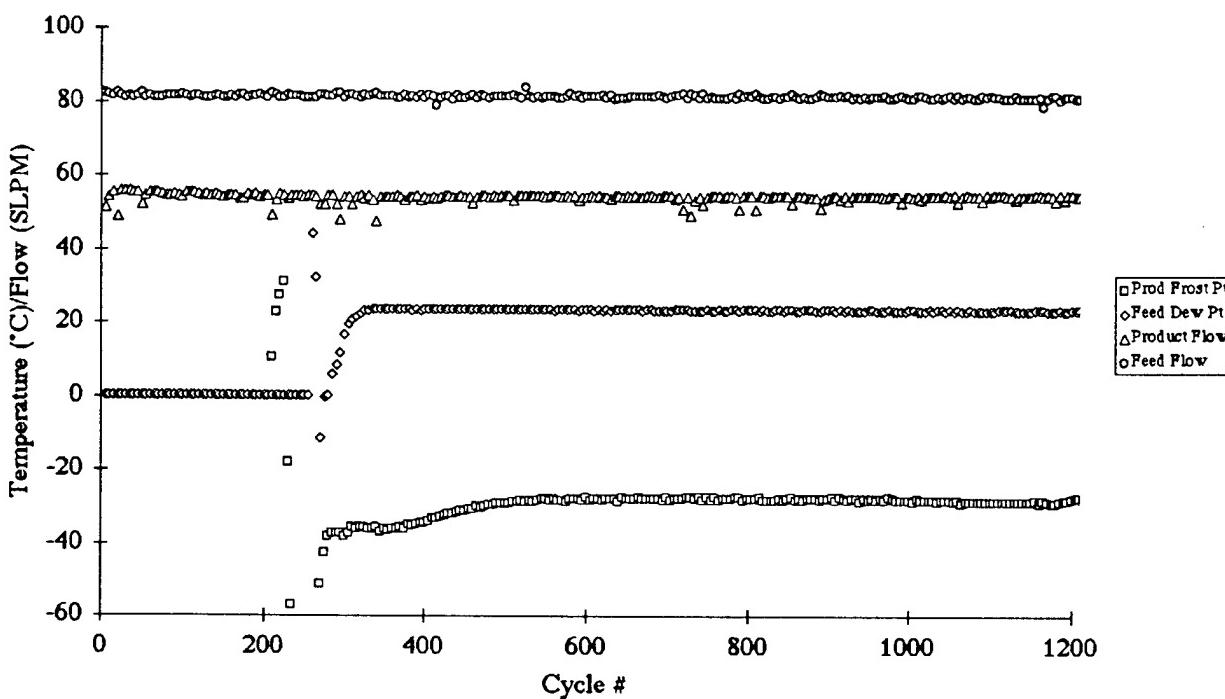
Figure 18. PSA Parameters (Feed and Product Flows/Dew Points), Expt-3-Layer_1/2/97
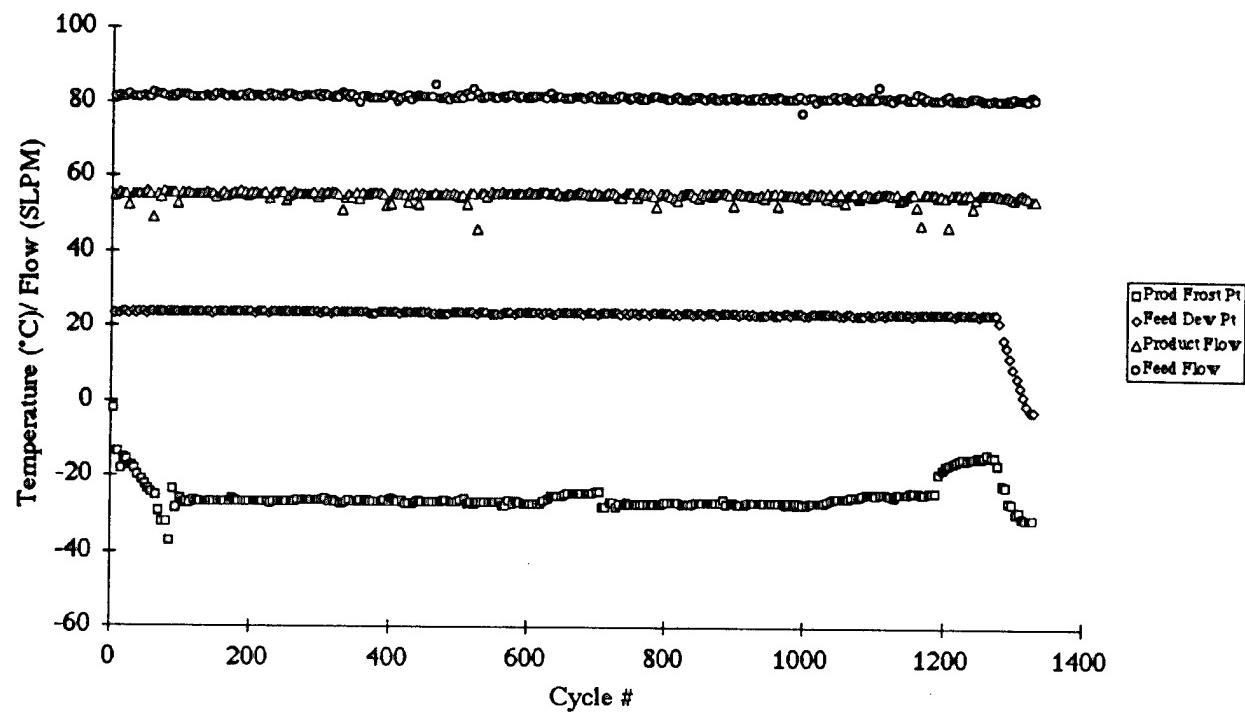
Figure 19. PSA Parameters (Feed and Product Flows/Dew Points), Expt-3-Layer_1/9/97
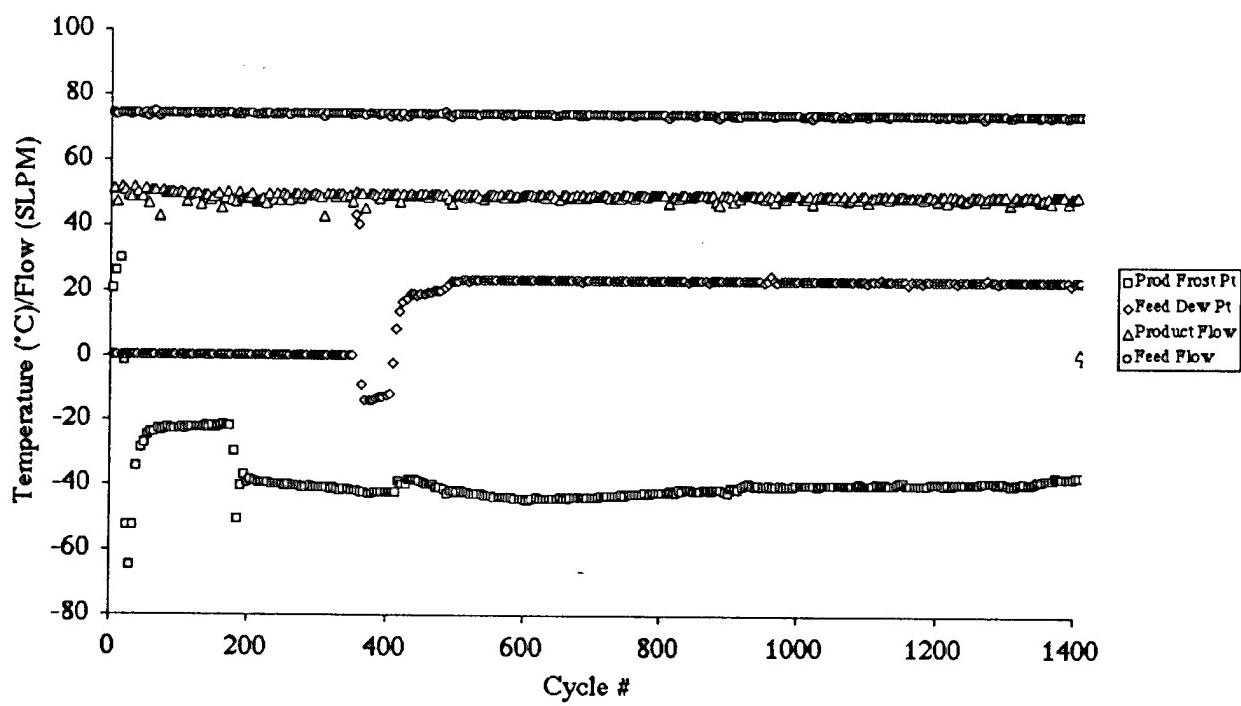

Figure 20. PSA Parameters (Feed and Product Flows/Dew Points), Expt-4-Layer_1/16/97
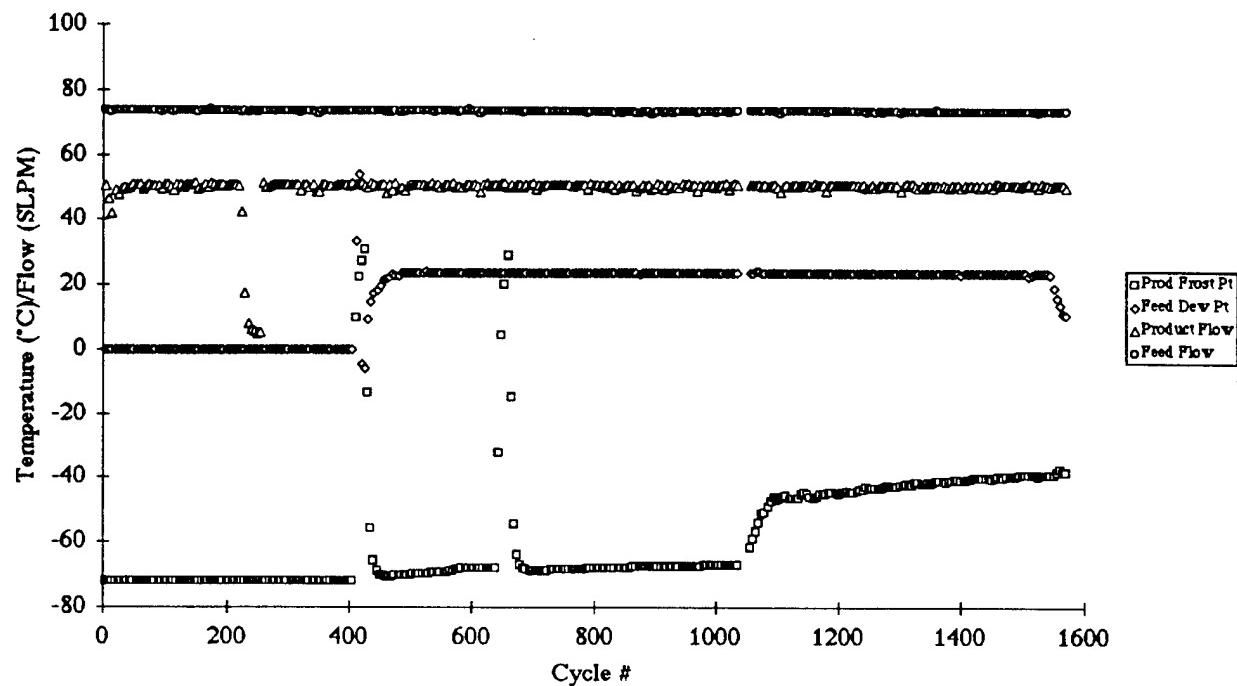
Figure 21. PSA Parameters (Feed and Product Flows/Dew Points), Expt-4-Layer_1/22/97
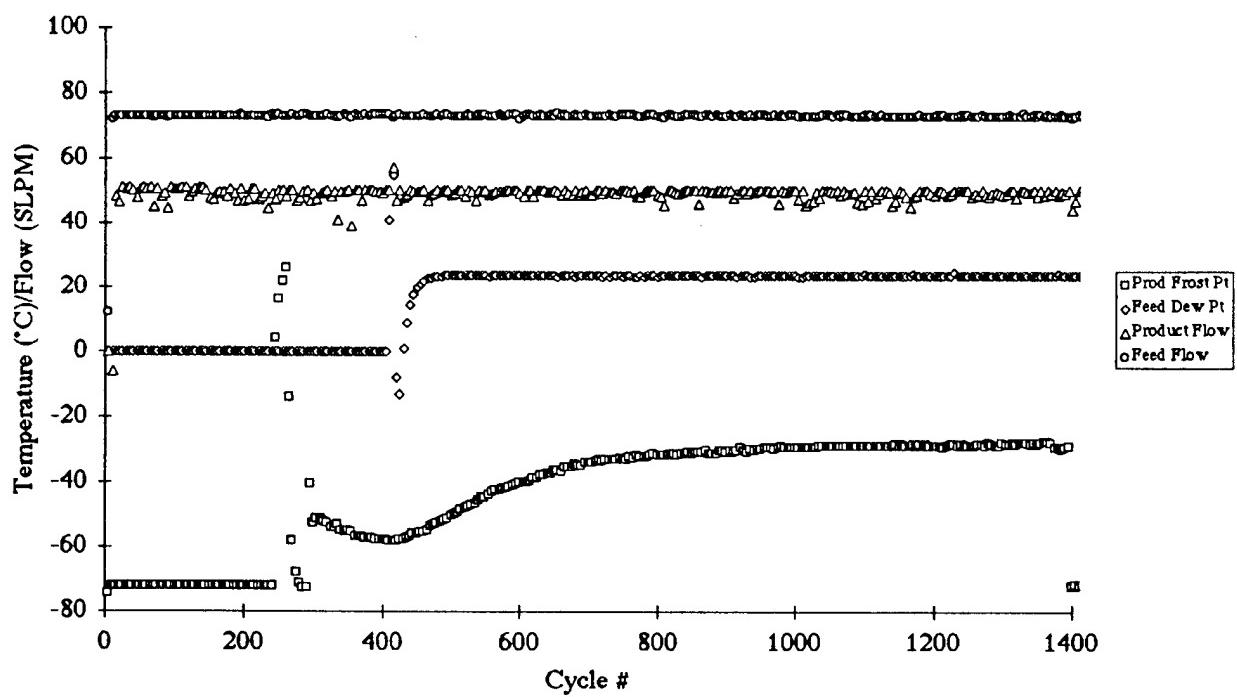

Figure 22. PSA Parameters (Feed and Product Flows/Dew Points), Expt-5-Layer_1/23/97
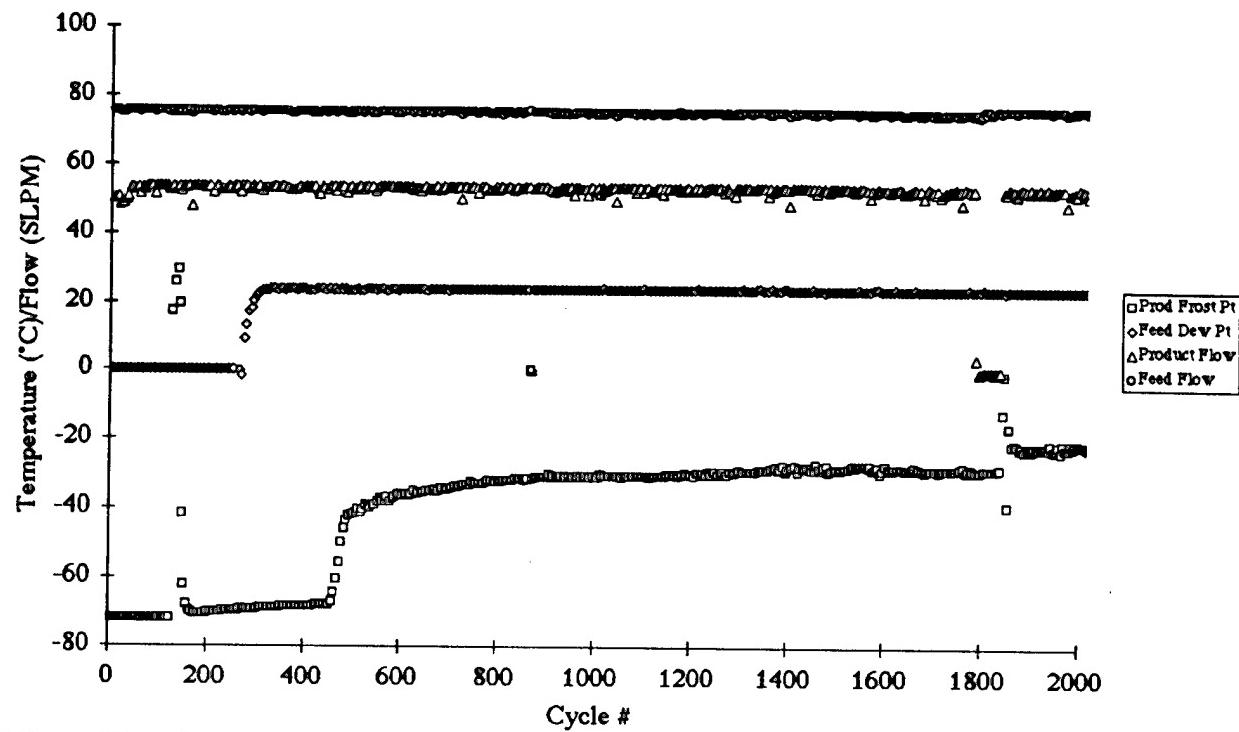
Figure 23. PSA Parameters (Feed and Product Flows/Dew Points), Expt-5-Layer_1/28/97
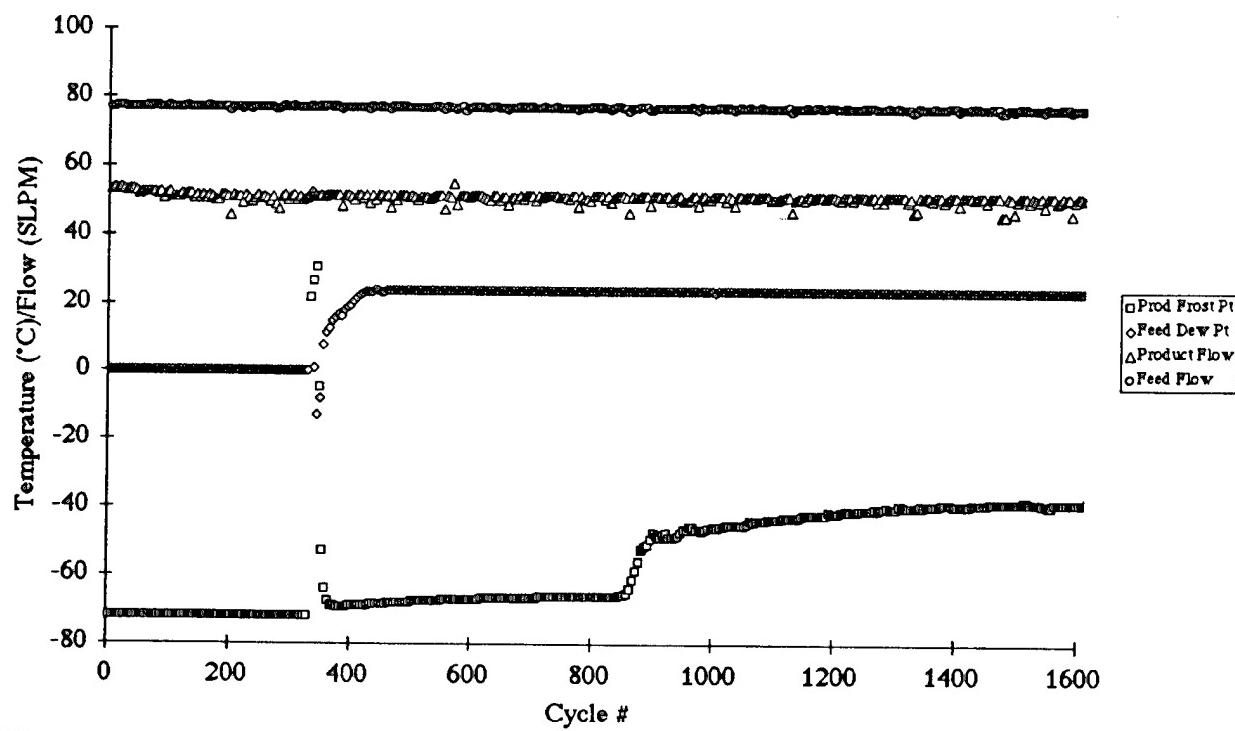
Figure 24. PSA Parameters (Feed and Product Flows/Dew Points), Expt-5-Layer_1/30/97

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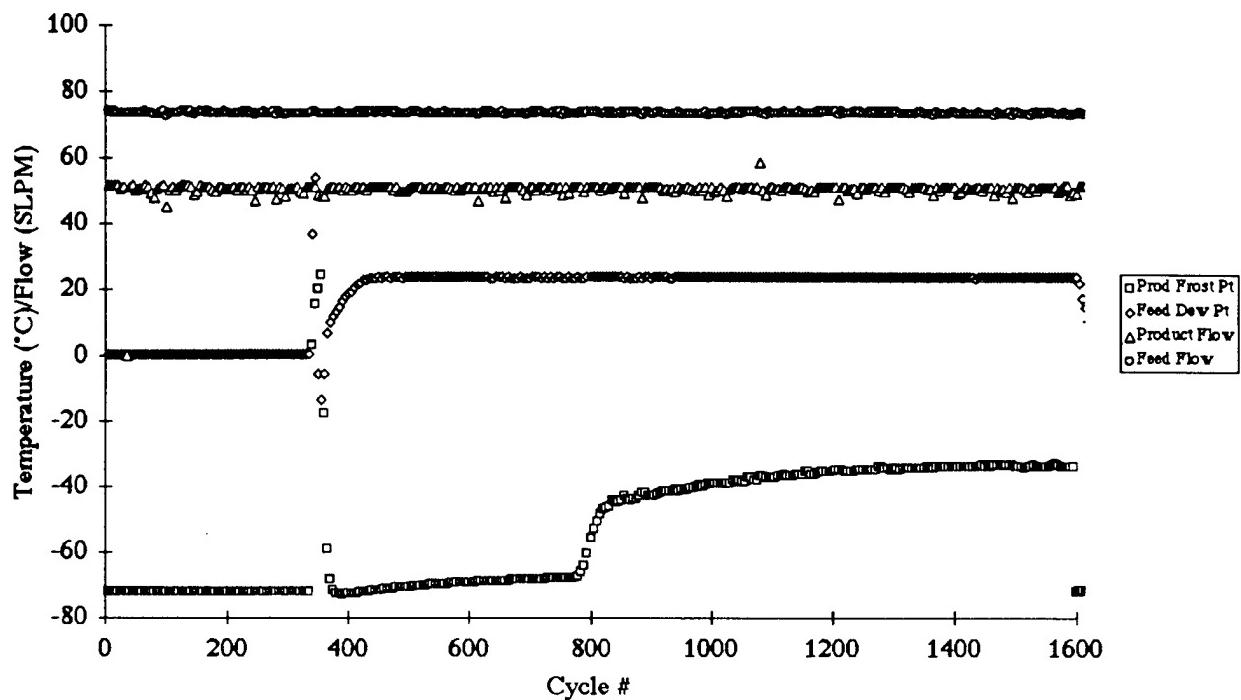
Figure 25. PSA Parameters (Feed and Product Flows/Dew Points), Expt-5-Layer_2/3/97
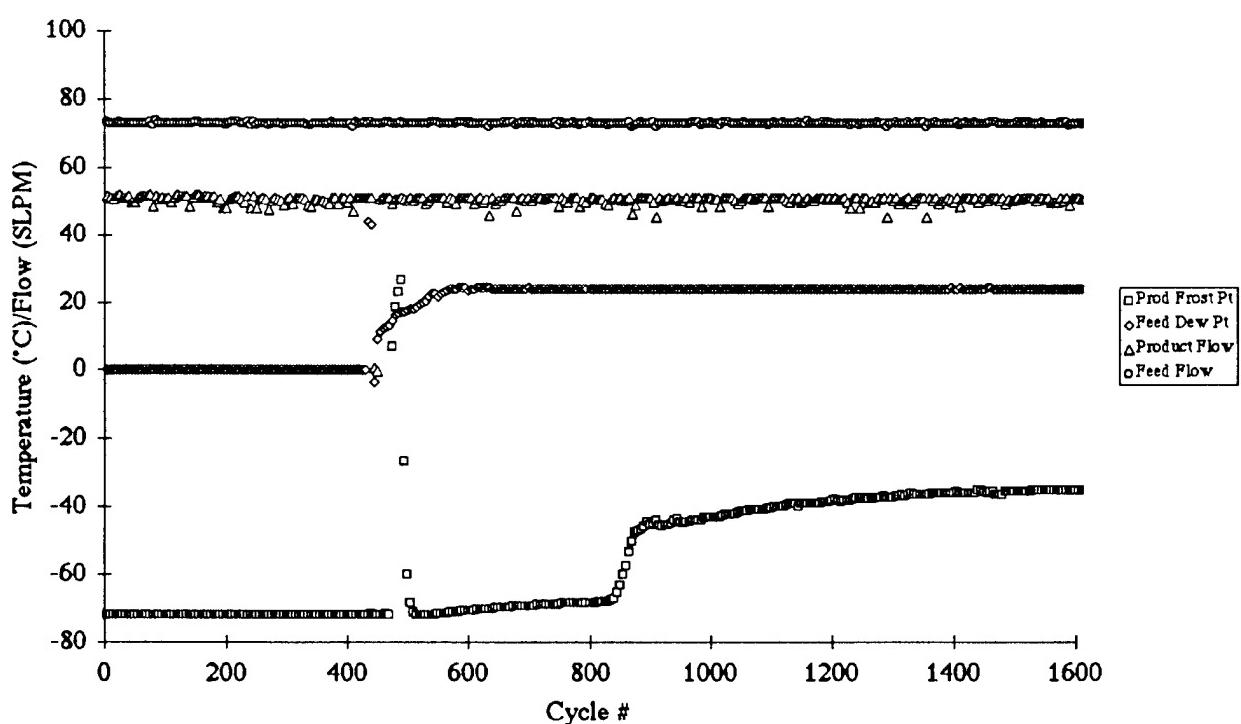
Figure 26. PSA Parameters (Feed and Product Flows/Dew Points), Expt-5-Layer_2/5/97
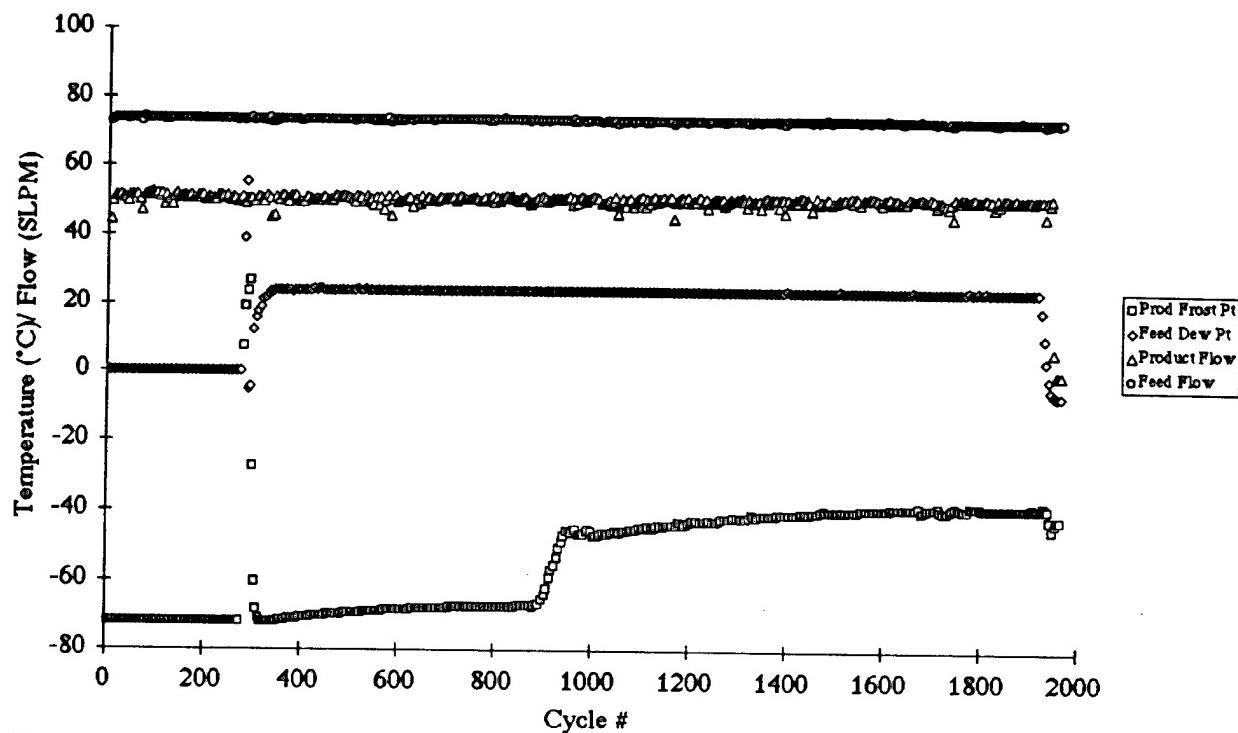
Figure 27. PSA Parameters (Feed and Product Flows/Dew Points), Expt-5-Layer_2/6/97
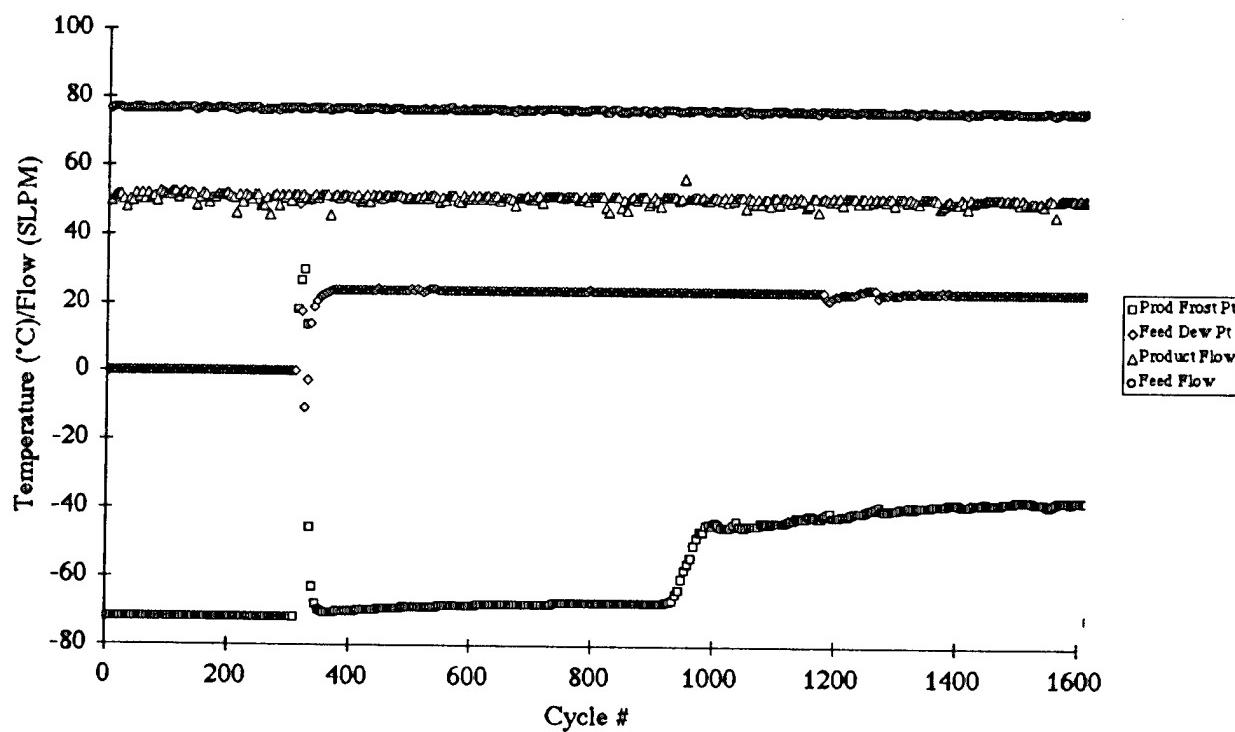
Figure 28. PSA Parameters (Feed and Product Flows/Dew Points), Expt-5-Layer_2/10/97
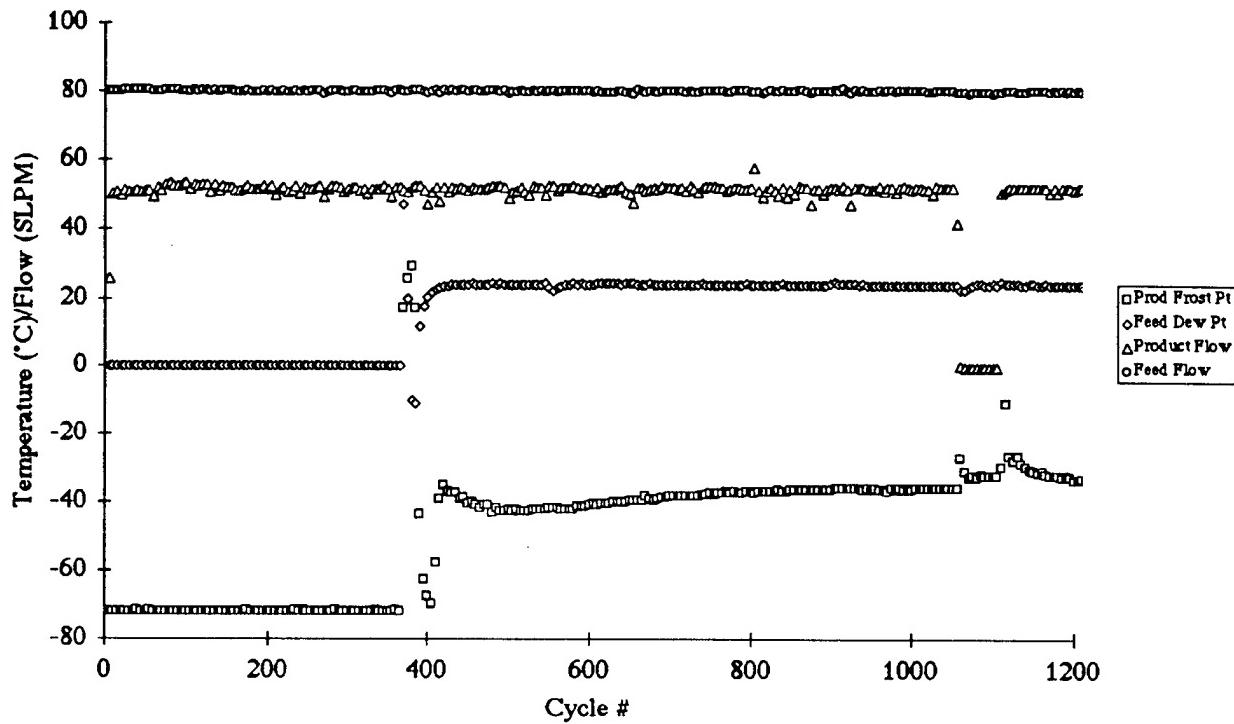
Figure 29. PSA Parameters (Feed and Product Flows/Dew Points), Expt-5-Layer_2/13/97
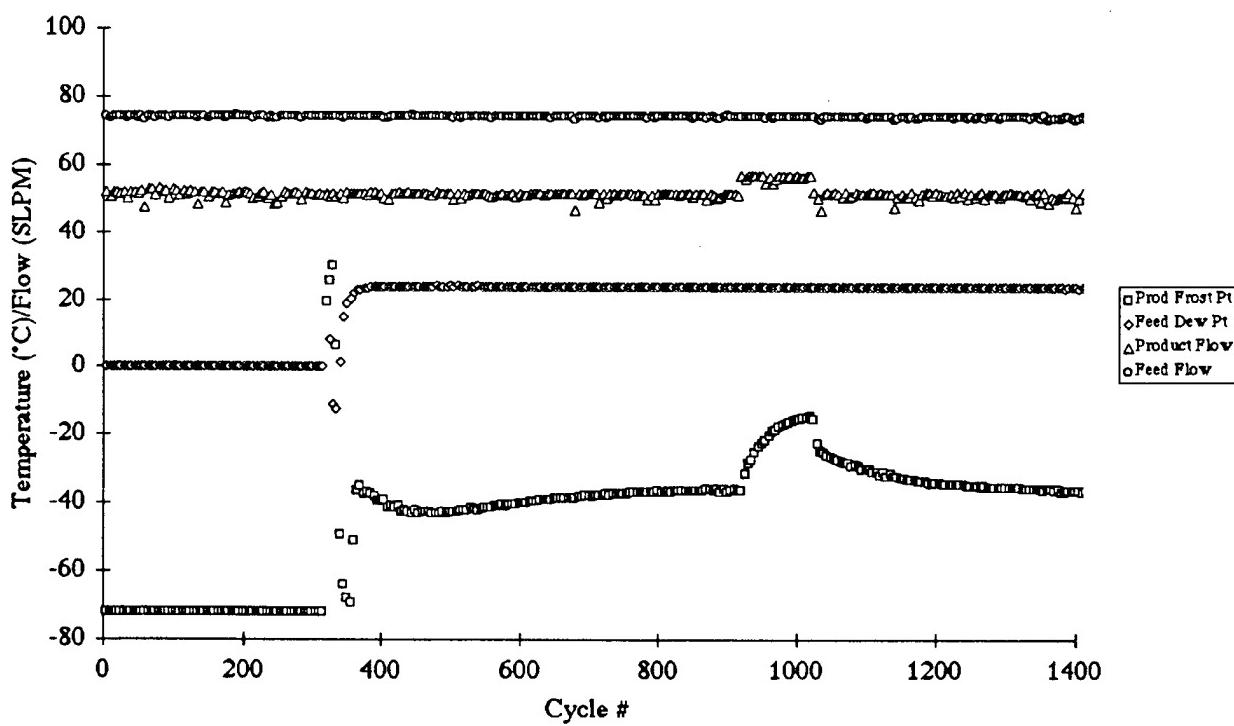
Figure 30. PSA Parameters (Feed and Product Flows/Dew Points), Expt-5-Layer_2/18/97
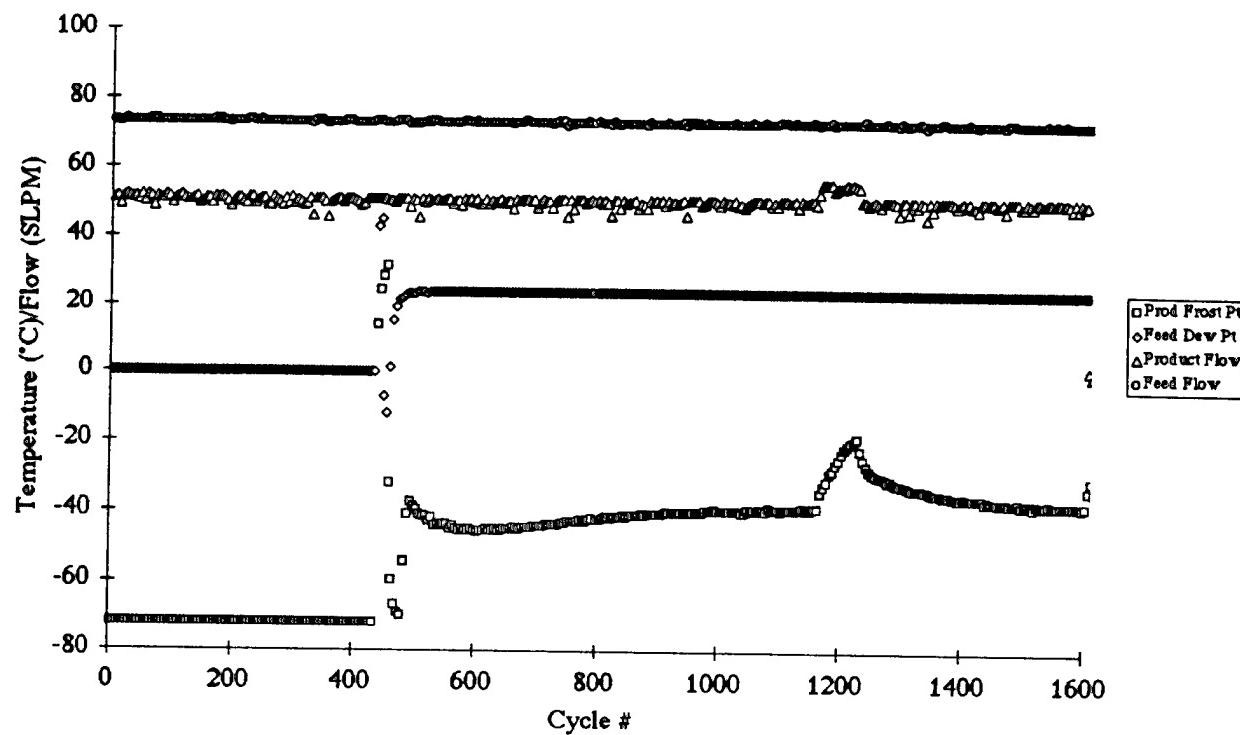
**Figure 31.** PSA Parameters (Feed and Product Flows/Dew Points), Expt-5-Layer_2/19/97
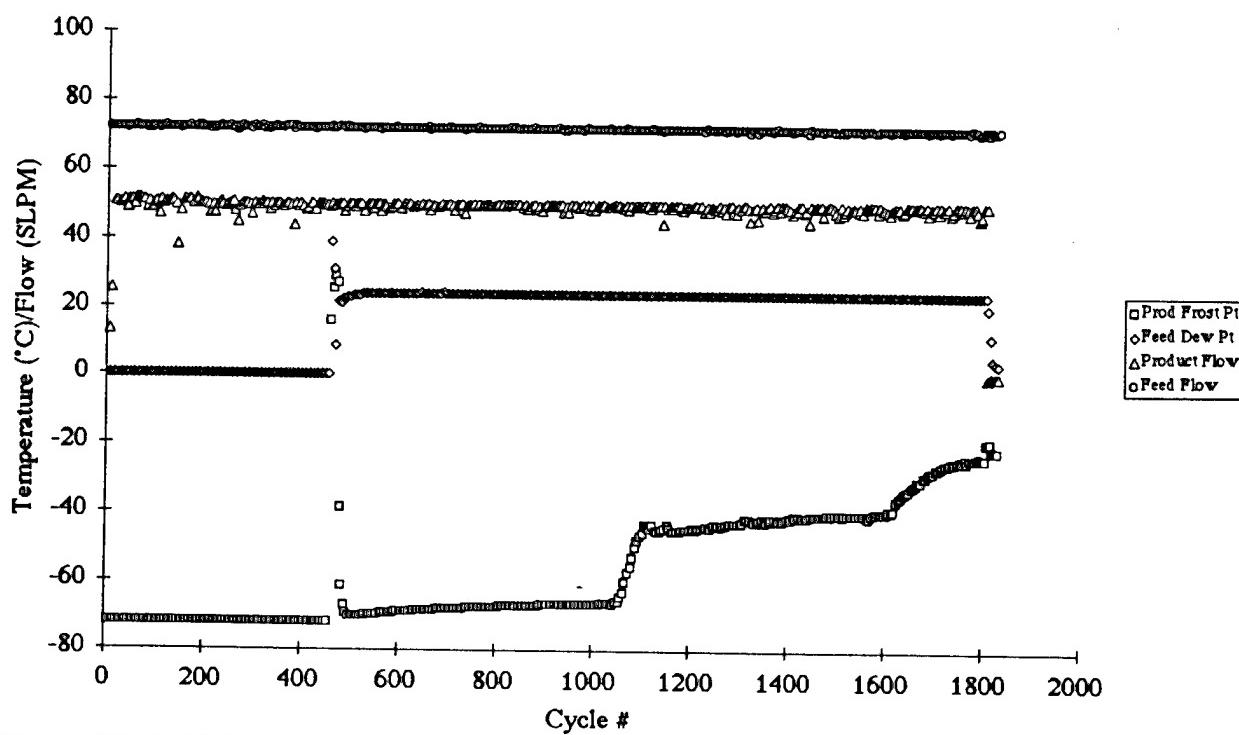
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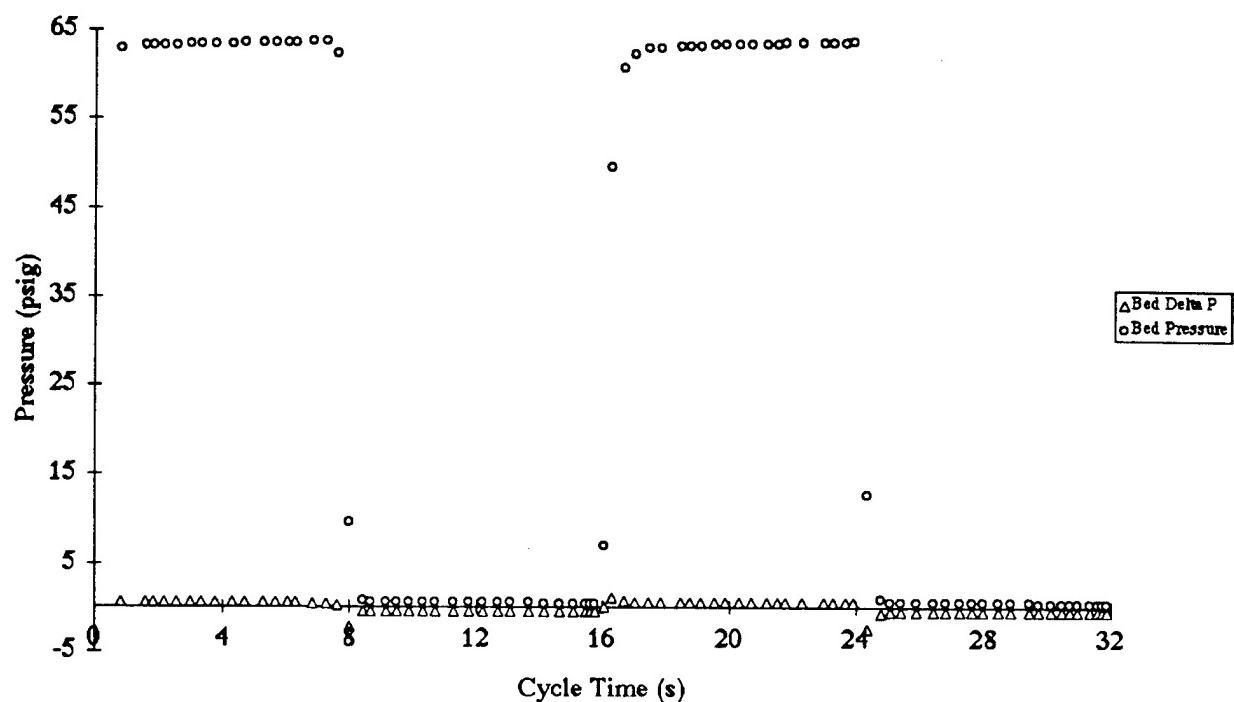
Figure 33. PSA Transient Pressure Profiles For Cycle Time of 16 sec, Expt-1/2/97
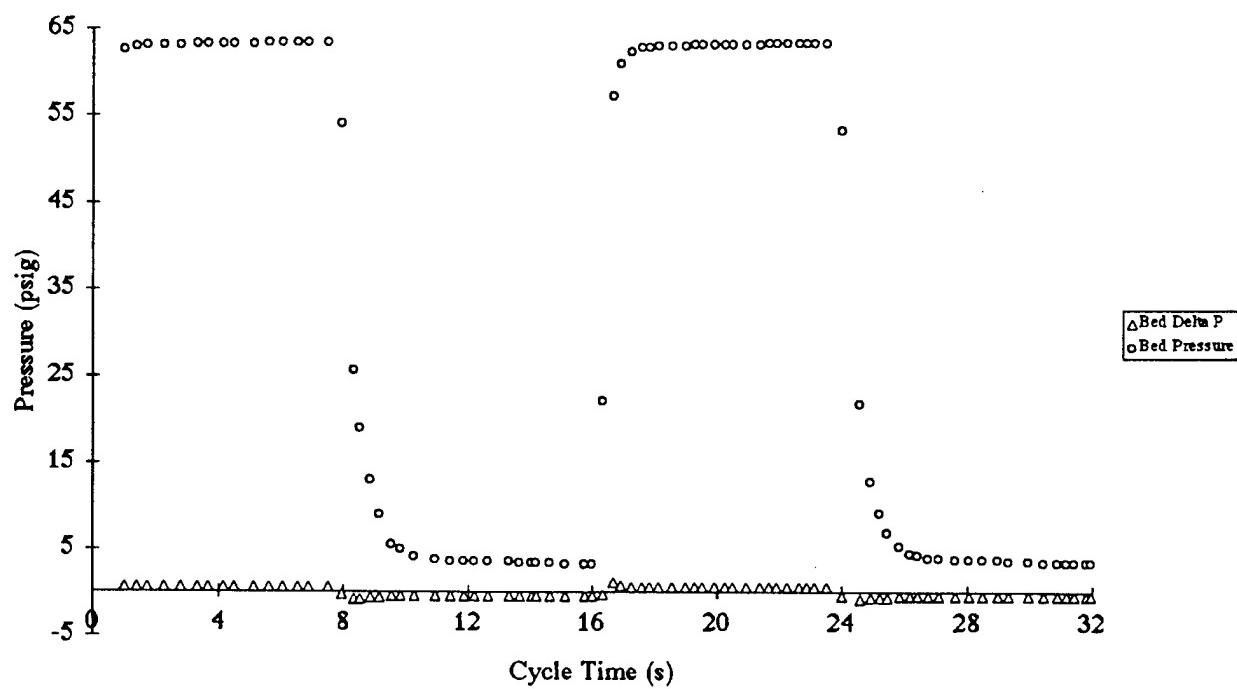
Figure 34. PSA Transient Pressure Profiles For Cycle Time of 16 sec, Expt-1/9/97
With a Purge Back Pressure of 3.5 psig

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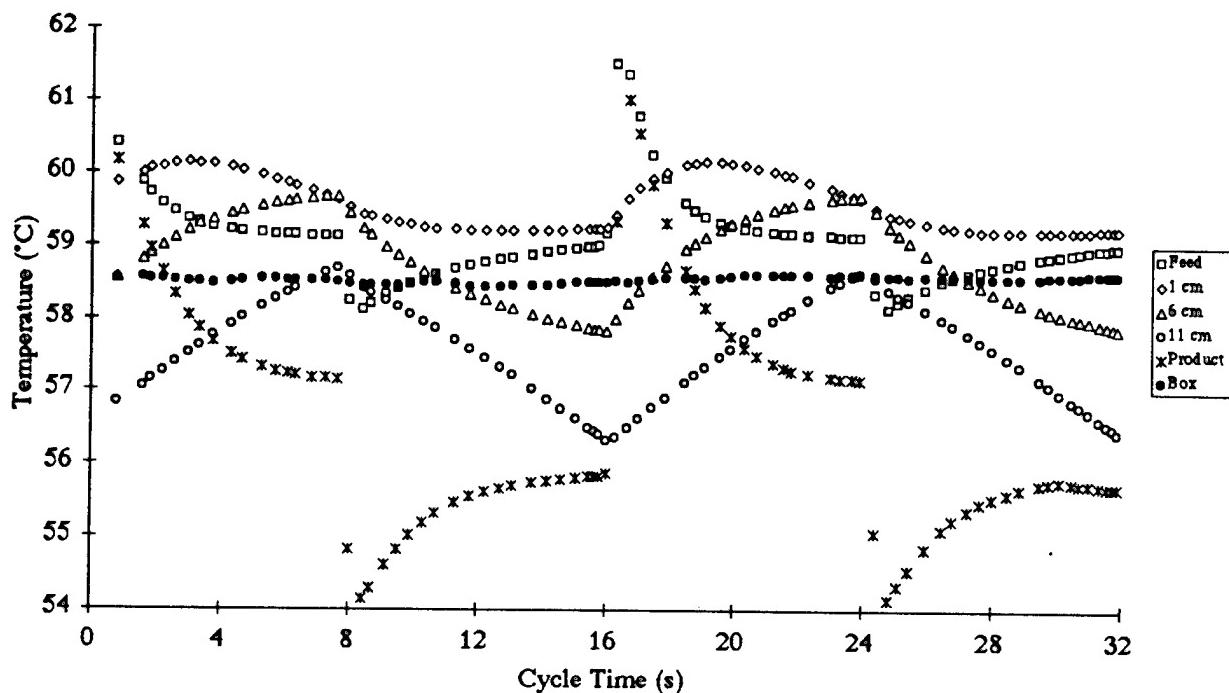
Figure 35. PSA Transient Temperature Profiles at Cycle 200, Before Water Added, Expt-1/2/97
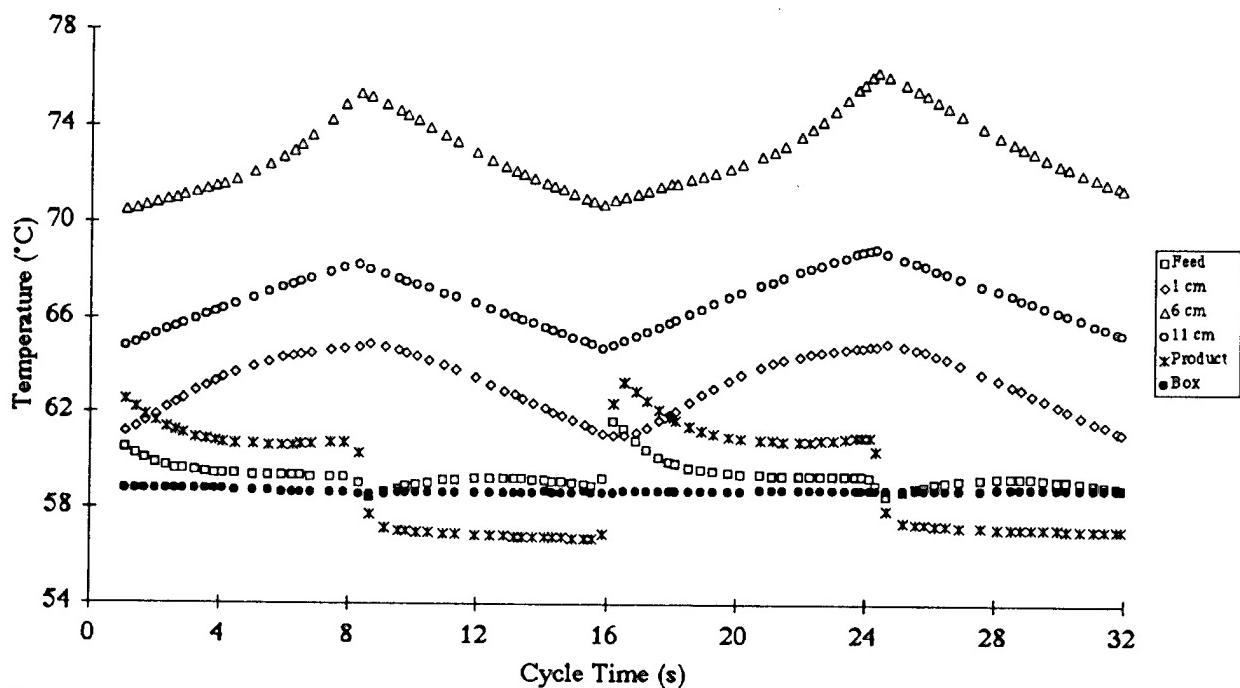
Figure 36. PSA Transient Temperature Profiles at Cycle 300, After Water Addition, Expt-1/2/97
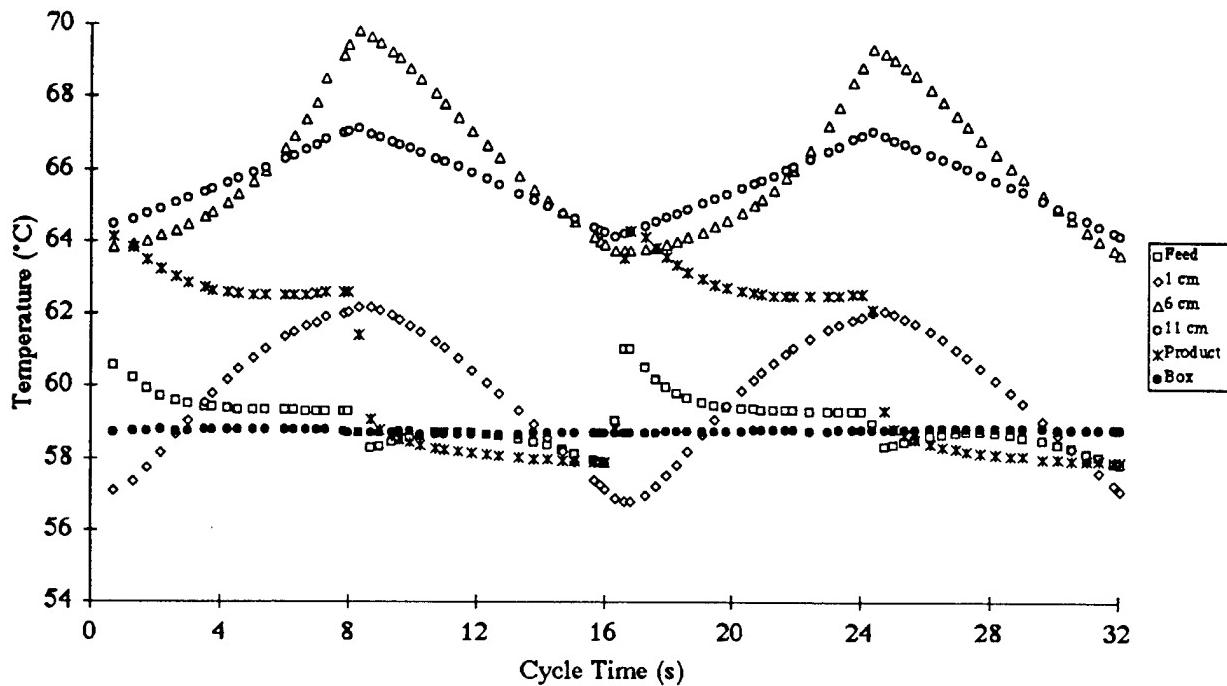
Figure 37. PSA Transient Temperature Profiles at Cycle 350, After Water Addition, Expt-1/2/97
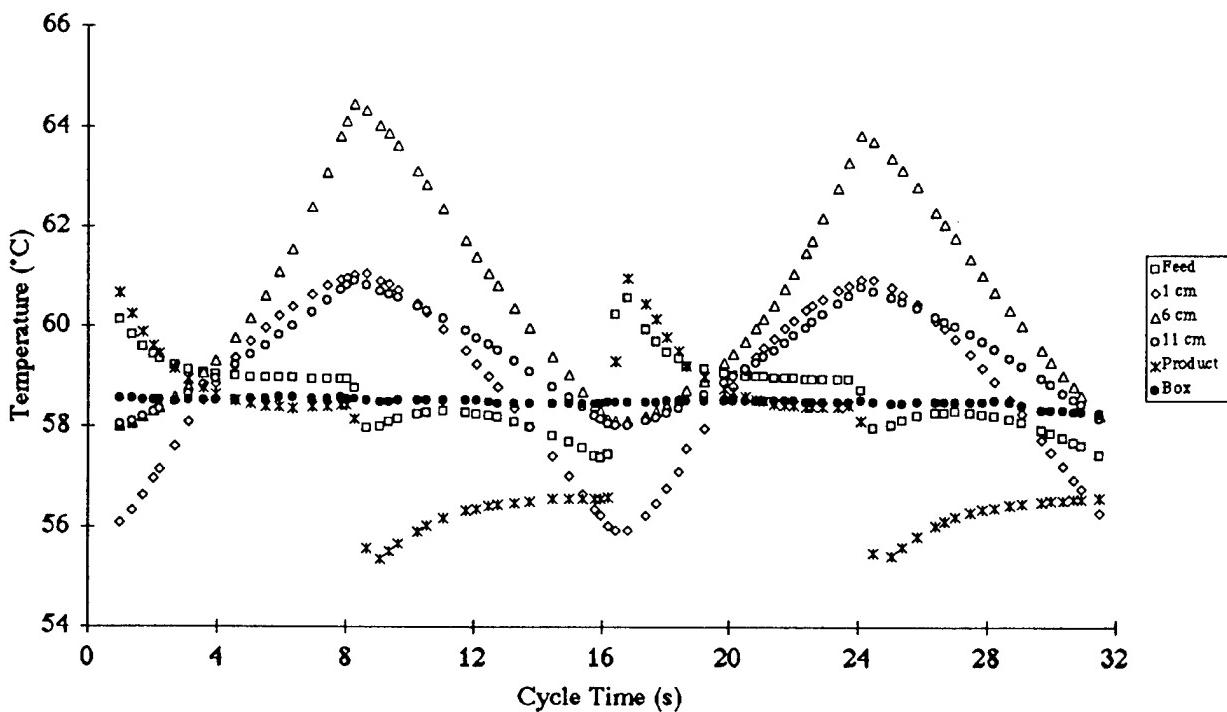
Figure 38. PSA Transient Temperature Profiles at Cycle 1000, Periodic State, Expt-1/2/97

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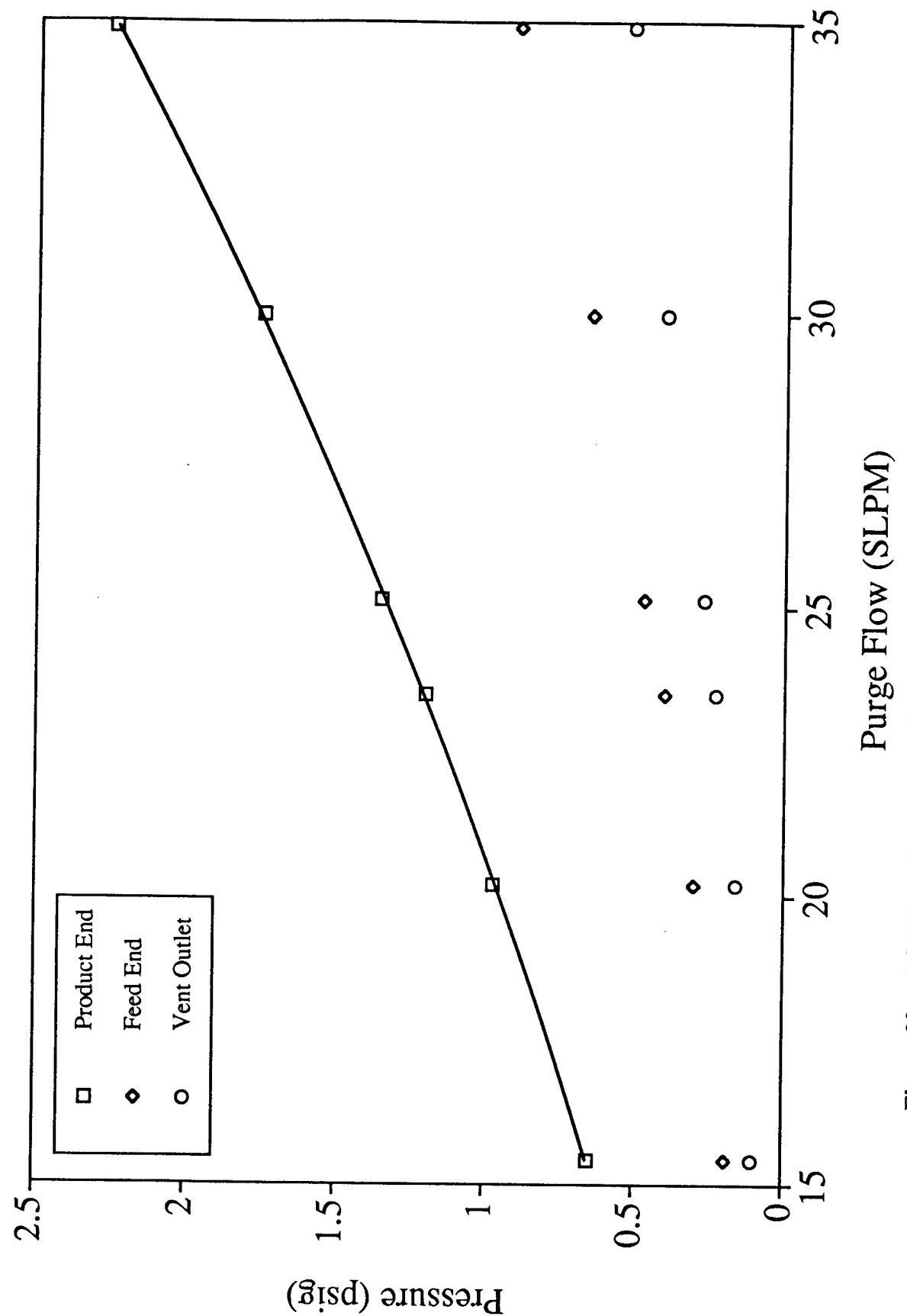
Figure 39. PSA Purge Pressure Profiles Versus Purge Flowrate for 3-Layer Bed

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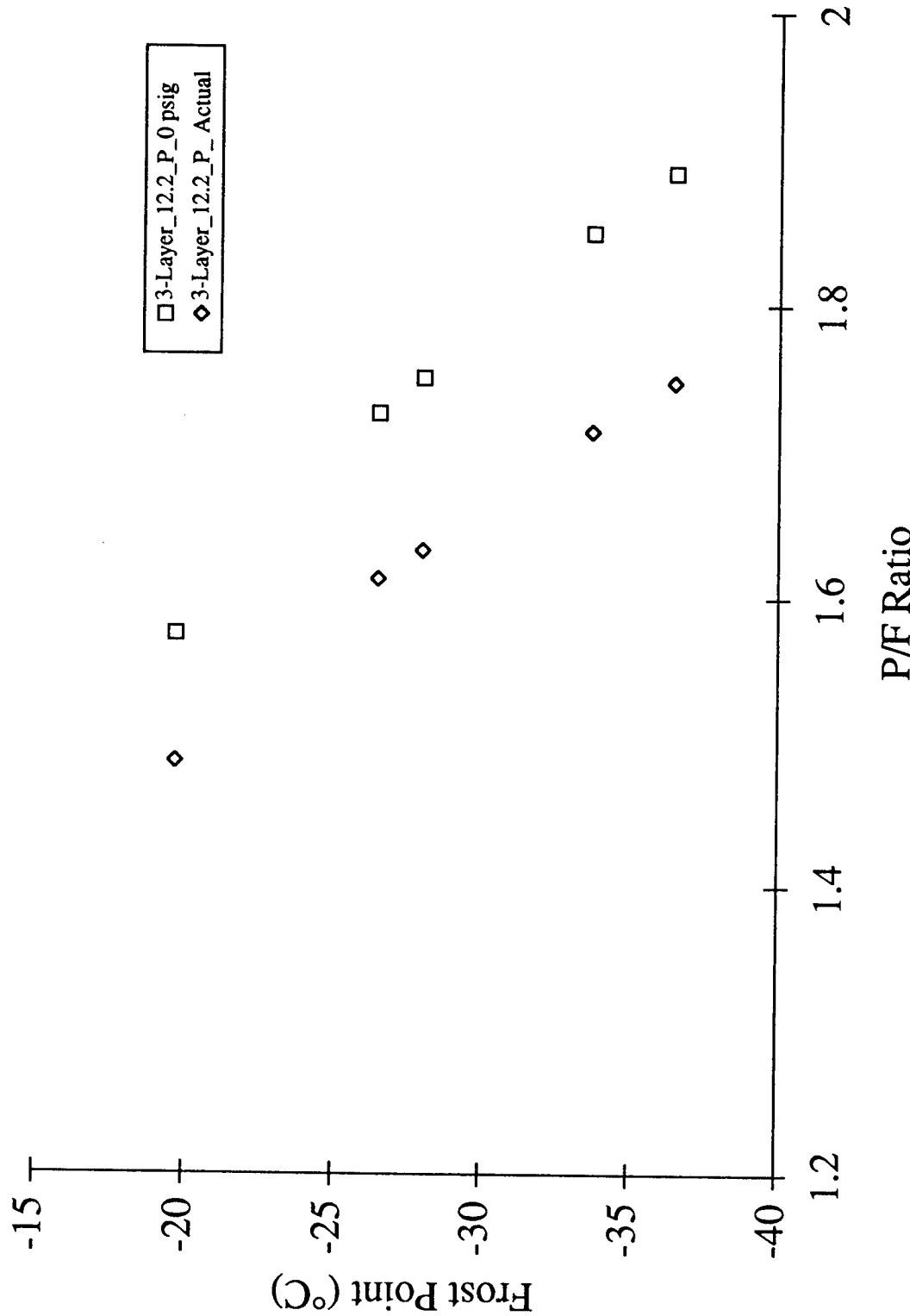
Figure 40. Frost Point *vs* P/F Ratio of 3-Layer Labscale PSA Beds, Case 2 Simulation of 12.2" Full-Scale Beds Effect of Purge Pressure and Purge Flowrate on the P/F Ratio (Uncorrected for Blowdown Step)
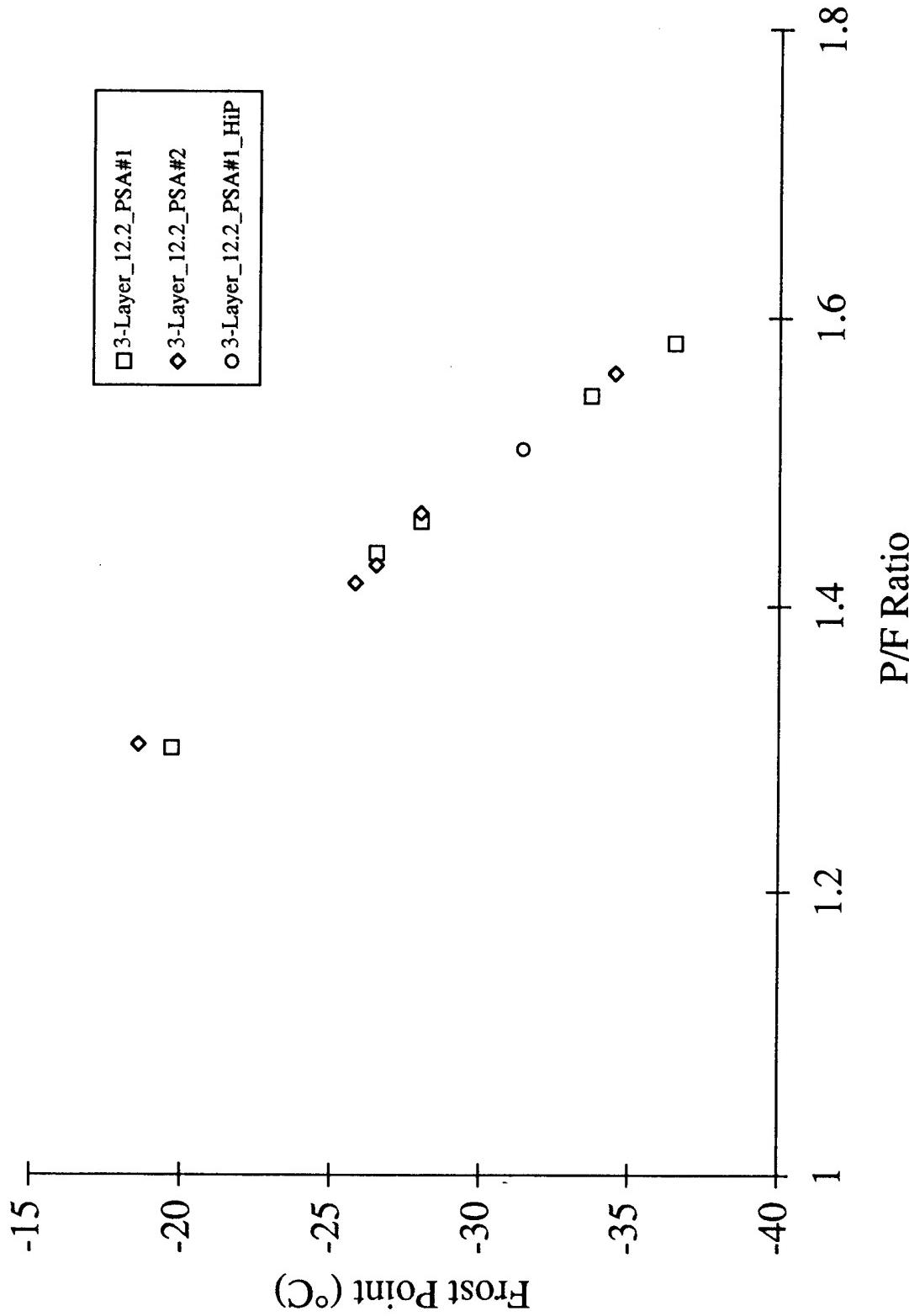
Figure 41. Frost Point vs P/F Ratio of 3-Layer Labscale PSA Beds, Case 2 Simulation of 12.2" Full-Scale Beds P/F Ratio Corrected for Blowdown and Pressurization of PSA Beds
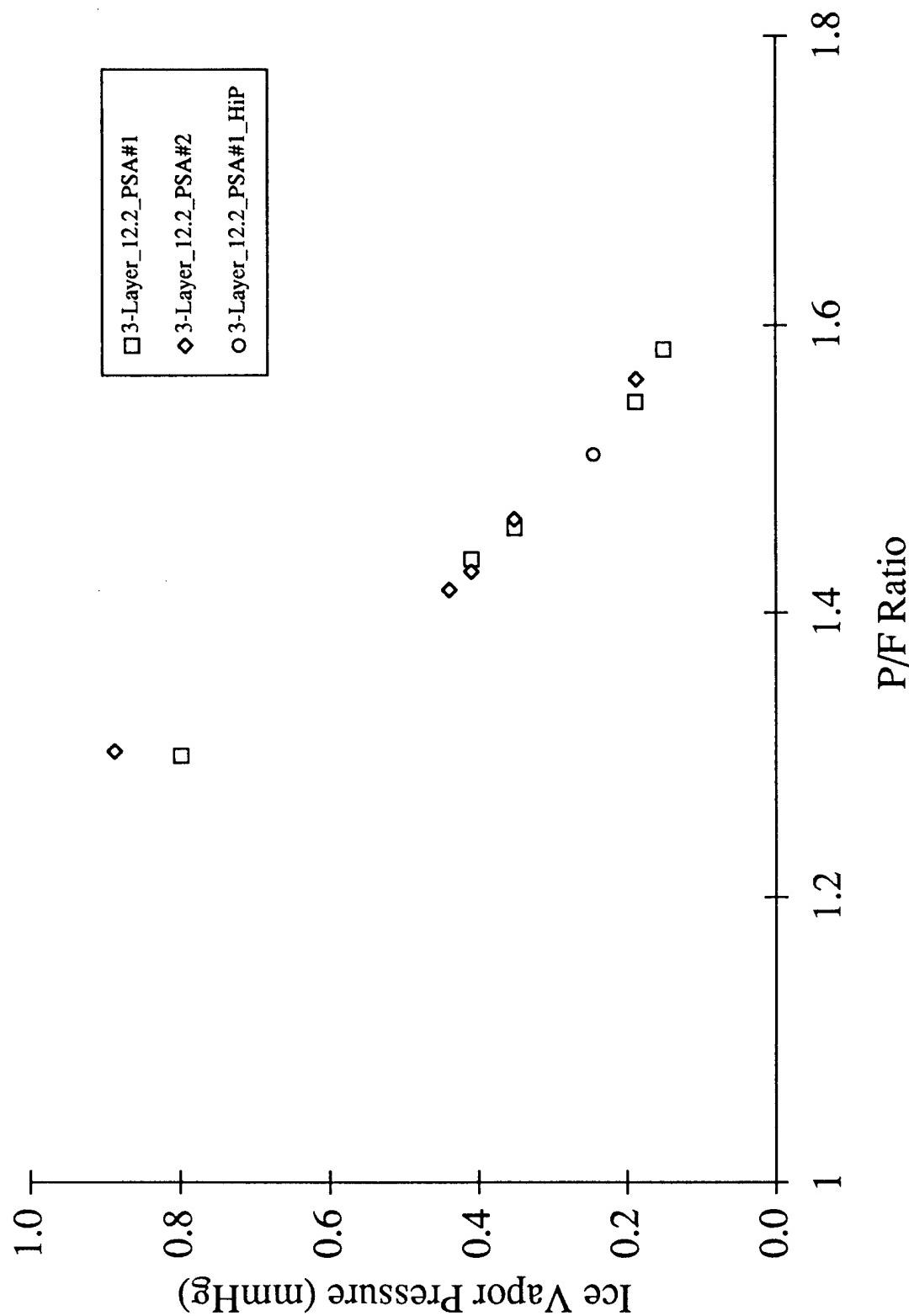
Figure 42. Ice Vapor Pressure vs P/F Ratio of 3-Layer Labscale PSA Beds, Case 2 Simulation of 12.2" Full-Scale Beds P/F Ratio Corrected for Blowdown and Pressurization of PSA Beds

Table 1. Adsorbent Nomenclature and Properties

Adsorbent Name	Common Name	Mesh Size	Adsorbent Appearance	Manufacturer
Alumina F-200	Alumina	7x14	Spherical White	Coastal Chemical
Coarse Silica Gel 40	Silica Gel 40	10x12	Granular Transparent	Grace-Davison Chemical
Fine Silica Gel 40	Silica Gel	18x25	Granular Transparent	Grace-Davison Chemical
Mol Sieve 13X (HP-PSA02)	13X	16x20	Spherical Beige	UOP
Activated BPL Carbon	Carbon BPL	12x30	Granular Black	Calgon Carbon

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Table 2. PSA Labscale Bed Configurations: a) 13X, b) 3-Layer, c) 4-Layer, and d) 5-Layer

a) 13X Molecular Sieve PSA Bed Configuration

Adsorbent Name	Layer End	Mesh Size	Adsorbent Mass (g)	Adsorbent Depth (cm)
13X	All	16x20	43.0	12.0

b) 3-Layer PSA Bed Configuration

Adsorbent Name	Layer End	Mesh Size	Adsorbent Mass (g)	Adsorbent Depth (cm)
Alumina	Feed	7x14	9.3	2.5
Silica Gel 40 (Coarse)	Middle	10x12	27.8	7.0
13X	Product	16x20	9.3	2.5

c) 4-Layer PSA Bed Configuration

Adsorbent Name	Layer End	Mesh Size	Adsorbent Mass (g)	Adsorbent Depth (cm)
Alumina	Feed	7x14	9.3	2.5
Silica Gel 40 (Coarse)	Middle-F	10x12	18.3	4.5
Silica Gel 40 (Fine)	Middle-P	18x25	9.3	2.0
13X	Product	16x20	11.6	3.5

d) 5-Layer PSA Bed Configuration

Adsorbent Name	Layer End	Mesh Size	Adsorbent Mass (g)	Adsorbent Depth (cm)
Alumina	Feed	7x14	9.3	2.5
Silica Gel 40 (Coarse)	Middle-F	10x12	18.3	4.5
Silica Gel 40 (Fine)	Middle	18x25	9.3	2.0
13X	Middle-P	16x20	5.6	1.5
BPL	Product	12x30	6.0	2.5

Table 3. PSA Labscale Conditions: a) Case 2 ICD Conditions and b) Labscale Conditions

a) Case 2 ICD Conditions

Feed Flow (lb/min)	Purge Flow (lb/min)	Prod Flow (lb/min)	Water Mixing Ratio (gr/lb)	Feed Dew Pt (°C@1atm)	Pressure (psia)	Temp (°F)	P/F (Flow)
32.3	10.5	21.8	137.8	24.7	78	134	0.325

b) Labscale Basis (1" Beds) for Case 2 ICD Conditions*

Full-Scale Bed Diameter	Feed Flow (SLPM)	Purge Flow (SLPM)	Prod Flow (SLPM)	Water Flow (ml/min)	Feed Dew Pt (°C@1atm)	Pressure (psia)	Temp (°C)	P/F (Flow)
10.8"	97.3	31.6	65.7	2.47	24.7	78	56.7	0.325
12.2"	76.2	24.8	51.4	1.93	24.7	78	56.7	0.325
12.5"	72.6	23.6	49.0	1.85	24.7	78	56.7	0.325

* Flowrates from full-scale beds are scaled to 1" labscale beds to attain same superficial fluid velocity

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Table 4. PSA Labscale Experiments: a) 13X Bed, b) 3-Layer Bed, and c) 4/5-Layer Bed

a) 13X Molecular Sieve PSA Bed Experiments

Expt #	Expt Name	Experiment Summary	ICD Case	Full-Scale Diameter (")	Product Dew Pt (°C)
1_13X	13X_8/28/96	Standard Conditions	2	10.8"	-10.0
2_13X	13X_9/4/96	Standard Conditions	2	10.8"	-8.2
3_13X	13X_9/10/96	Standard Conditions	2	10.8"	-10.2
4_13X	13X_9/11/96	Standard Conditions	2	10.8"	-11.1
5_13X	13X_9/13/96	Standard Conditions	2	10.8"	-3.4
6_13X	13X_9/16/96	Standard Conditions	2	10.8"	-9.1
7_13X	13X_9/19/96	Standard Conditions	2	10.8"	-8.6

Table 4 (con'd): PSA Labscale Experiments: a) 13X Bed, b) 3-Layer Bed, and c) 4/5-Layer Bed

b) **3-Layer PSA Bed Experiments**

Expt #	Expt Name	Experiment Summary	ICD Case	Full-Scale Diameter (")	Product Dew Pt (°C)
1_3-Layer	Al_Si_13X_10/4/96	Standard Conditions	2	12.2"	-34.5
2_3-Layer	Al_Si_13X_10/16/96	Standard Conditions	2	12.2"	-31.0
3_3-Layer	Al_Si_13X_10/17/96	Standard Conditions	2	12.2"	-29.5
4_3-Layer	Al_Si_13X_10/17/96	2 LPM Less Product	2	12.2"	-40.0
5_3-Layer	Al_Si_13X_10/17/96	1 LPM Less Product	2	12.2"	-37.0
6_3-Layer	Al_Si_13X_10/18/96	2 LPM More Product	2	12.2"	-22.0
7_3-Layer	Al_Si_13X_10/23/96	Standard Conditions	2	12.2"	-29.4
8_3-Layer	Al_Si_13X_10/23/96	2 LPM Less Product	2	12.2"	-37.9
9_3-Layer	Al_Si_13X_10/24/96	Standard Conditions	2	12.2"	-28.6
10_3-Layer	Al_Si_13X_10/24/96	1 LPM Less Product	2	12.2"	-30.9
11_3-Layer	Al_Si_13X_10/24/96	2 LPM More Product	2	12.2"	-20.7
12_3-Layer	Al_Si_13X_1/2/97	Std Conditions/Inverted Beds	2	12.2"	-30.9
13_3-Layer	Al_Si_13X_1/9/97	Std Conditions/Inverted Beds	2	12.2"	-29.9
14_3-Layer	Al_Si_13X_1/9/97	1 psi Purge Pressure/Inverted Beds	2	12.2"	-26.6
15_3-Layer	Al_Si_13X_1/9/97	3.5 psi Purge Pressure/Inverted Beds	2	12.2"	-14.5

Table 4 (cont): PSA Labscale Experiments: a) 13X Bed, b) 3-Layer Bed, and c) 4/5-Layer Bed

c) **4/5-Layer PSA Bed Experiments:**

Expt #	Expt Name	Experiment Summary	ICD Case	Full-Scale Diameter (")	Product Dew Pt (°C)
1_4-Layer	Al_Si_Si_13X_1/16/97	Standard Conditions	2	12.5"	-43.1
2_4-Layer	Al_Si_Si_13X_1/22/97	Standard Conditions	2	12.5"	-42.1
3_5-Layer	Al_Si_Si_13X_BPL_1/23/97	13X Removed/BPL Added	2	12.5"	-31.4
4_5-Layer	Al_Si_Si_13X_BPL_1/28/97	25% LessAlumina/13X Added	2	12.5"	-33.5
5_5-Layer	Al_Si_Si_13X_BPL_1/30/97	50% Less Alumina/13X Added	2	12.5"	-41.6
6_5-Layer	Al_Si_Si_13X_BPL_2/3/97	15% Less Silica/13X Added	2	12.5"	-37.3
7_5-Layer	Al_Si_Si_13X_BPL_2/5/97	25% Less Silica/13X Added	2	12.5"	-38.4
8_5-Layer	Al_Si_Si_13X_BPL_2/6/97	3g Less Silica&Alumina/13X Added	2	12.5"	-42.6
9_5-Layer	Al_Si_Si_13X_BPL_2/10/97	50% Less Al&15% Less Si/13X Added	2	12.5"	-40.5
10_5-Layer	Al_Si_Si_13X_BPL_2/13/97	3g Less Silica&Alumina/13X Added	2	12.5"	-40.0
11_5-Layer	Al_Si_Si_13X_BPL_2/18/97	3g Less Silica&Alumina/13X Added	2	12.5"	-40.0
12_5-Layer	Al_Si_Si_13X_BPL_2/19/97	50% Less Al&15% Less Si/13X Added	2	12.5"	-42.1
13_5-Layer	Al_Si_Si_13X_BPL_2/27/97	50% Less Al&15% Less Si/13X Added	2	12.5"	-43.2
14_5-Layer	Al_Si_Si_13X_BPL_2/27/97	Expt #13 with 2 psi Purge Pressure	2	12.5"	-26.0

Table 5. PSA Optimization Summary: Experiment Number, Adsorbent Mass, Breakthrough Time, and Product Frost Point

Adsorbent	Expt1 Expt2 (Std)	Expt3 + (BPL)	Expt4 -25% (Al)	Expt5 -50% (Al)	Expt6 -15% (Si)	Expt7 -25% (Si)	Expt8 -3g (Si/Al)	Expt9 (Opt)	Expt10 -3g (Si/Al)	Expt11 -3g (Si/Al)	Expt12 (Opt)	Expt13 0 psi (Opt)	Expt14 2 psi (Opt)
PSA Bed	4-Layer	5-Layer	5-Layer	5-Layer	5-Layer	5-Layer	5-Layer	5-Layer	5-Layer	5-Layer	5-Layer	5-Layer	5-Layer
Alumina	9.3	9.3	7.0	4.7	9.3	9.3	6.3	4.7	6.3	6.3	4.7	4.7	4.7
Silica Gel 40 (Coarse)	18.3	18.3	18.3	18.3	15.6	13.7	15.3	15.6	15.3	15.3	15.6	15.6	15.6
Silica Gel 40 (Fine)	9.3	9.3	9.3	9.3	9.3	9.3	9.3	9.3	9.3	9.3	9.3	9.3	9.3
13X	11.6	5.6	7.9	10.2	8.3	10.2	11.6	12.9	11.6	11.6	12.9	12.9	12.9
BPL Carbon	0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0
Total Bed Mass (g)	48.5	48.5	48.5	48.5	48.5	48.5	48.5	48.5	48.5	48.5	48.5	48.5	48.5
B.T. Time (hr)	2.8	0.8	1.1	2.1	1.8	1.6	2.7	2.9	-	-	-	-	-
Product Frost Pt (°C)	-39.5 -38.5	-28.5	-30.5	-38.0	-34.0	-35.0	-39.0	-37.0	-36.5	-36.5	-38.5	-39.5	-23.5

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